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VAPORIZING AND ENDOTHERMIC FUELS FOR ADVANCED ENGINE APPLICATION

Part II. Studies of Thermal and Catalytic Reactions, Thermal Stabilities, and Combustion Properties of Hydrocarbon Fuels

A.C. Nixon, G.H. Ackerman, L.E. Faith, H.T. Henderson, A.W. Ritchie, L.B. Ryland

Shell Development Company, A Division of Shell Oil Company

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Air Force Aero Propulsion Laboratory
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FOREWORD

This report was prepared by Shell Development Company, Emeryville, California, under U.S. Air Force Centract No. AF 33(615)-3789. The contract was initiated under Project No. 3048, Task No. 304801. The work was administered under the direction of the Aero Propulsion Laboratory, Mr. H. L. Iander, Project Engineer, AFPL.

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A. C. Nixon was principal investigator and project supervisor for Shell Development Company. The professional staff participating in the investigation was comprised of: G. H. Ackerman, L. E. Feith, H. T. Henderson, A. W. Ritchie, and L. B. Ryland.

This report was submitted by the authors

This technical report has been reviewed and is approved.

Arthur V. Churchill, Chief Fuels, Lubrication and Hazards Branch Support Technology Division

ABSTRACT

Investigation of the feasibility of using endothermic reactions of hydrocarbons to augment the latent and sensible heat of fuels for cooling engines operating under a high mach number regime is continuing. The literature continues to maintain the desirability and feasibility of producing vehicles with hypersonic flight speeds; some areas of advantage for hydrocarbons are suggested.

The dehydrogenation of Decalin over a platinum/Al₂O₃ catalyst has been studied extensively on a laboratory scale covering the temperature region up to 1200°F and pressures to 10 atms, in both once through and differential systems, in order to provide kinetic data for the construction of a mathematical model. Studies on the thermal reaction of SHELLDYNE and its hydrogen treated derivative indicate that the hydrogen treating increased the stability by a factor of 1500 (the reactivity of SHELLDYNE-H is about the same as that of Decalin).

Production of catalysts under our catalyst development program have turned up a number of the 536 catalysts examined that are more active than four standard Pt/Al₂O₃ catalyst, but no breakthrough in either activity or cost has been achieved. Further testing has shown that catalysts which demonstrate improved activity with MCH do the same with Decalin and has confirmed the observation that improved catalyst stability is associated with small pore size. Our efforts to reduce the heat transfer and pressure drop problems inherent in a bed catalyst by applying the cataly. To the wall of the heat exchange tube have met with some success. Studies on molecular and dispersed catalysts are underway. A pulse reactor to be used in this study has been successfully operated with MCH.

Heat transfer studies in a simulated single tube fuel system have confirmed the mathematical model for the catalytic dehydrogenation of MCH up to a heat flux of 600,000 Ptu/hr/sq ft. They also indicate the necessity for catalysts of higher activity and stability such as are available from our catalyst development program. Heat transfer studies with nonreactive cooling to be applied with a regenerative ramjet indicate maximum temperature limits of 1350°F for tube wall and 1150°F for the fluid. A maximum heat flux of 85 x 10° Btu/hr/sq ft has been achieved to date. Satisfactory correlations of the Dittus/Roelter type have been developed for the super critical region but are less satisfactory in the critical and subcritical regions.

In studying the effect of environment on the thermal stability of Decalin at 600°F, it was found that exposure to high surface areas of iron, copper and chromium had a deleterious affect which could be largely controlled with MDA.

SHELLDYNE-H was found to have a satisfactorily short ignition delay in shock tube studies compared to other hydrocarbon systems. An interesting "double delay" behavior was observed with SHELLDYNE itself. The thermodynamic and transport properties of trans-Decalin, SHELLDYNE and JP-7 and a bibliography of recent literature of interest are included.

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VAPORIZING AND ENDOTHERING FUELS FOR ADVANCED ENGINE APPLICATION

Introduction

The objective of this study is to provide the information necessary for specifying fuels which will be capable of providing cooling and propulsion for engines powering aircraft in the speed range above lach 3. The fuel will provide cooling by giving up its latent and sensible heat and by undergoing endothermic reactions before it is fed into the engine as vaporized fuel. Practically, this could be in the temperature range up to about 1400°F. In order for the fuel to function in this manner, it must have excellent thermal stability up to the temperature at which reaction occurs and also in the post-reaction portion of the heat exchanger, to avoid fouling problems. Work under early Air Force contracts served to establish many of the parameters which obtain in delineating the boundaries of the problem. Work was done under our previous contract¹⁻³ to define more closely the advantages and limitations for the application of hydrocarbon fuels. In that contract it was intended to develop specifications for a fuel or fuels which could be utilized for advanced engine application and to design methods and equipment for testing the properties of such a fuel.

In order to allow precise definition of the fuel, we studied various problems that could arise in several parts of the fuel-combustion system. These included thermal stability problems which could originate in the fuel tanks or in the various metering devices and fuel lines; deposition or coking problems which could affect the efficiency of heat exchanger-reactor devices and catalysts, or plug fuel nozsles; and combustion parameters which could affect the design or operation of the combustion chambers. In order to provide a sound basis for the selection or rejection of fuels, we endeavored to relate the various phenomena observed to the physical and chemical properties of the fuels studied.

The problem areas and approaches used were broken down in the following manner: we improved a previously designed coker apparatus to permit it to be used to study the thermal stability of possible fuels and components at temperatures up to 900°F. We studied possible thermal and catalytic reactions in laboratory scale equipment in order to test the reactivity of fuels and the suitability of selected catalysts. The heat sinks available in the hydrocarbons tested were calculated from thermodynamic properties of the reactants and products. A fuel system simulation test rig (FSSTR) was constructed and used to provide data on hydrocarbon systems. A computer program for simulating the behavior of a packed bed reactor was modified to accept and correlate the results obtained in the fuels system simulator. The subsonic combustion properties of selected fuels and reaction products were observed in a small scale combustor while the ignition-delay behavior of the same fuels and products was studied in a single-diaphragm shock tube to give an indication of supersonic combustion properties.

¹⁾ See References.

> Studies done under the previous contract indicated the general feasibility of the endothermic reaction approach, particularly the utilization of catalytic dehydrogenation reactions. Our best results have been achieved with the platinum/alumina-methylcycloherane combination. With this combination, the possibility of achieving the original conceptual goal of 2000 Btu per pound of fuel total heat sink seems possible. The importance of restricting the oxygen content to very low levels to reduce heat exchanger problems was also indicated. Operations with the fuel system simulation test unit (FESTR) have provided valuable data for heat exchanger design calculations and demonstrated the possibility of high space velocity and long catalyst life with the MCH system. Limitation of thermal cracking of hydrocarbons to a relatively low heat sink of about 300 Btu per pound due to hydrogen transfer reaction was demonstrated. The mathematical model for the cylindrical axial flow reactor was found to be adequate to the extent of its present development. The combustion studies suggest that the possibilities of burning the proposed feed materials and the products of their dehydrogenation under both subsonic and supersonic combustion conditions are premising.

Under the present contract we are continuing and extending the work done under the previous contract with some changes in emphasis. We are continuing to survey the pertinent literature and will issue bibliographies from time to time. We will continue to consider various feed materials which might be useful in this application and assess the probability of their being successful candidate materials. Such candidates are screened in our small scale equipment for reactivity and effect on catalyst life and their thermal stability under heat exchange conditions. Successful candidate materials are tested with improved catalysts and also under larger scale conditions as represented by our fuel system simulation test rig.

In the previous contract only a limited number of catalysts, selected for their probable activity, were tested with a variety of feed materials. The reactions of interest in that program included dehydrogenstion, dehydrocyclization and depolymerization. In the present program we are conducting an extensive catalyst development program for new catalysts for these types of reactions. This involves the small scale preparation of a wide variety of catalysts in which catalytic elements (e.g., transition metals) are deposited on substrates and modified by a variety of noncatalytic elements such as, for example, the alkalies, alkaline earths, and halogens. Other catalysts are prepared containing metallic oxides and acidic sites. Such catalysts are tested initially in a small scale apparatus ("micro scale catalyst test reactor", MICTR) which allows rapid screening with standard feed materials such as MCH, n-heptane and tetraisobutylene. In addition to the attempt to prepare superior conventional type catalysts in which catalytic materials are mounted on substrate granules, attempts are being made to prepare nonconventional catalysts in which the catalytic material is mounted in a special way designed to minimize pressure drop, or is previously dispersed in the feed material, or is formed by decomposition in the heated zone. Such nonconventional catalysts are tested with MCH in a variety of equipment prior to being used with other feed materials developed as a result of the program mentioned above.

The computerized mathematical model mentioned above has been carried to the point where good representation of the FSSTR experimental results was possible with different size tubes and catalyst dispositions and

variable heat flux and temperatures along the tube length. Difficulty was encountered in representing different tube lengths, LHSV's and heat fluxes but this appears to have been resolved. The program will eventually include latent and sensible heat sinks both before and after the reaction zone, and the mating of a reactor-exchanger with a heat source such as a combustor or a leading edge. This latter step, of course, will require collaboration with engine manufacturers who are working on the combustor design part of the overall program.

Extension of the program towards an aircraft system requires that considerably more work be done on the supersonic combustion aspects of the problem. We are continuing our studies in the shock tube with high molecular weight fuels and their dehydrogenation products. Fuels utilized by engine companies may also be examined in the shock tube to obtain correlation with the results observed by them in their laboratory studies. We also contemplate completing our work on the examination of subsonic combustion in the small burner developed under the previous contract with the additional feature of obtaining quantitative data on the radiation emitted, as a function of fuel composition and burner conditions.

An important consideration in any system which attempts to use the fuel for cooling is the thermal stability of the fuel in the exchanger portion portions of the fuel system. In our previous contract we used the SD Coker for evaluating the thermal stability both of feed material and of products produced by both thermal and catalytic reactions. Examination of the products suffers from the serious deficiency that inevitably a time lapse and some handling has to occur, before the products of reaction are tested. We have therefore constructed a new piece of equipment under the present contract for establishing a standard test for both catalysts and fuels. This unit, called the Catalyst and Fuel Stability Test Rig (CAFSTR), permits simulation of the thermal environment and representative contact times all the way from the fuel tank to the engine inlet. Fuels will be tested using a standard catalyst, while catalysts will be tested using a standard fuel.

Specific support is also being furnished to contractors in the cooling program. This support consists of consultation with respect to problems encountered in the study programs, the furnishing of technical data required for the solution of design problems or for the carrying out of experimental investigations.

Summary

Work to bring our knowledge of the Decalin system up to that we have of MCH has continued. Because of the existence of two isomers in the feed and the two-step nature of the dehydrogenation, the Decalin system is considerably more complicated than MCH. Kinetic data has been obtained in diluted beds and under recycle conditions allowing for completion of a mathematical model for the system.

The thermal reactivity of the high density fuel, SHELLDYNE, and the effect of hydrogen treatment on this reactivity has been studied in our bench scale reactors. SHELLDYNE was studied over the temperature range 770 to 940°F at pressures up to 10 atms. The thermal reactivity increased with both temperature and contact time. In the all metal system used, SHELLDYNE was found to be quite reactive leading to coking at the higher contact times and temperatures; pressure had little effect. Hydrogen treating gave a much more thermally stable product as was evidenced in studies covering the temperature range 1022 to 1202°F and pressures to 10 atms. It is estimated that SHELLDYNE is 1500 times as reactive as the hydrogen-treated material. In both cases, the different isomers had different rates of reaction. SHELLDING H was found to be only slightly more reactive than Decalin. Testing of catalysts prepared under our catalyst development program has continued. The general procedure is to screen the catalysts rapidly with MCH as the test fluid, examining promising catalysts more extensively under bench scale conditions with MCH and if they are still favorable, utilize Decalin as a further test fluid. A group of six catalysts that appeared promising under beach scale conditions with MCH have now been tested further with Decalin. Out of this study has come a number of catalysts of improved activity and stability with both MCH and Decalin. Further evidence that the stability of the cutalyst is related inversely to the pore size of the support have been obtained. An additional five outslysts which had appeared promising under micro scale test conditions have been evaluated in bench scale reactor testing. On the basis of preliminary work two of the catalysts appear 12 and 16% more active than the standard cutalyst. Additional cutalysts have also been prepared and submitted to micro scale testing. The total number of catalysts prepared under our program or obtained from proprietory or commervial sources now stands at 536. During this period, the effect of neutralising the platinizing solutions with different acids has been investigated with some favorable effects noted. Although most of the catalysts prepared have less activity than the standard material, twelve new materials have been found this quarter which are more active. One interesting case of a bimetallic system has been found in which the synergistic effect of an inactive metal on a slightly active metal has been observed. Attempts have also been made to find catalysts which will be active for the dehydrocyclolisation of n-heptane to toluene to take advantage of the additional heat sink available in this way. Some moderately active catalysts were discovered but none of sufficient activity to serve the purpose. Cracking was a common side reaction.

More success has been had with attempts to develop a nonconventional configuration in which the catalyst is attached to the heat exchanger tube wall. Considerable experimental work has been done to develop ways of getting good bonding to the metal surface without destroying the good catalytic activity of the basic catalyst formulation and developing a technique for

applying the coatings to the inside surface of tubes. This seems to have been satisfactorily solved and a number of tubes were prepared with coating thicknesses ranging from 3.5 to 13 mils. In the area of developing particulate or molecularly dispersed catalysts, a number of possible compounds have been prepared or purchased. These will be tested out in two different types of apparatus, an injection autoclave and a pulse reactor. Preliminary work in the latter has turned up three model compounds that show some activity with MCH in this mode of application.

Work on the development of mathematical models to represent both reactive and nonreactive heat sink situations has continued. Difficulties in the development of a satisfactory model to represent the Decalin system in which dehydrogenation to tetralin and naphthalene occurs necessitated the acquisition of additional kinetic data under low conversion conditions. Also, the regenerative heat exchanger model for nonreactive cooling has required continual modification to represent experimental results. It now appears satisfactory in the super-critical region but not below.

Physical and transport properties for SHELLDYNE, a typical JP-7 and trans-Decalin have been calculated using the presently best regarded computation systems. These data are included in this report and have been transmitted to interested investigators. A 1500 gallon batch of Decalin has been purified by silica gel treatment.

The experimental study for very high heat flux conditions in support of the nonreactive cooling program has been continued using heat exchange systems made up of four- and six-inch lengths of 26.5 mil ID tubing in the modified FSSTR (the "mini-FSSTR"). Six sections have been constructed to date, all basically of the same design. These have been utilized in a series of tests with nitrogen, MCH, and water as the test fluids. Operating of flow rates up to 14 x 108 lbs/sq ft/hr, a heat flux of up to 8 million Btu/hr/sq ft was achieved with a maximum outlet temperature of about 900°F and a maximum inlet pressure of 1000 psi. Plugging and burn-out conditions were reached during some of these experiments, however, in only one case due to ooking.

Efforts to develop a simple and reproducible method of rating deposits on CAFSTR tubes has been continued. Methods favored include solvent removal of deposits, combustion and electron reflection. A large group of selected solvents were tested for their effects on deposits formed from Decalin and jet fuel in the ASIM coker, heating the solvent at 100°C for one hour. The only promising solvent found was dimethylformsmide which appeared to be capable of removing substantially all the deposit although two treatments are required. Some results on other methods of rating have been obtained.

The SD-coker has been modified to maintain the feed side of the system at system pressure so that the Zenith pump utilized serves merely as a metering device, thus avoiding the occurrence of pump wear. This system seems to be performing quite satisfactorily. Rydrogen treating SHELIDINE improved its stability substantially. Examination of the effect of powdered metals in Decalin revealed the combination Fe/MDA are be very deleterious.

Indications of the supersonic combustion behavior of three possible high density fuel components have been obtained by measuring ignition delays

in the single disphragm shock tube at temperatures to 2500° f and 25 psia pressure. The order of decreasing ignition delay found was dimethano-Decalin = SHELLDYNE, octane, SHELLDYNE H. All fuels had about the same energy of activation of combustion (40 kcal). SHELLDYNE showed a peculiar "double-delay" behavior wich so far remains inexplicable.

Considerations Affecting Applications

An interesting paper has recently been published by Ferri⁴) (Review of Scramjet Propulsion Technology) which, although directed towards the use of IH₂ fuel, and the combustion process rather than cooling, has some pertinent points. He points out the importance of good cooling since the maximum pressure rise across the combustor in the absence of separation depends strongly on the wall temperature in the region of separation. He notes that the most severe cooling and heat requirements occur in the burner. Good burner design requires that the values of both the heat that must be removed and the maximum heat transfer coefficient should be minimized but at the same time the engine wall thickness must be kept within practical limits. His remarks emphasize the necessity to extend our knowledge of cooling techniques to the upost in order to develop cooling systems of great flexibility that will not unduly constrain the engine designer.

An interesting application of the endothermic reaction principle can be made in the case of ablative thermal protective systems. This would involve incorporating into the material, a mixture of a high boiling naphthene and a finely divided supported, or molecularly dispersed, dehydrogenation catalyst. As the material heated up as the vehicle entered the atmosphere, one of the early reactions that would take place would be the dehydrogenation of the naphthene to an arcmatic and hydrogen with the resulting ejection of the latter from the solid or liquid layer. As explained by E. W. Ungar⁵) the heat transfer rate at the gas interface is substantially reduced from the usual value by gas injection. He noted:

"Injection of gas into the boundary layer cools the gas adjacent to the surface and thickens the layer, thus reducing heat transfer rates. The effectiveness of the injected gas in reducing heat transfer increases as the molecular weight of the injected gas is decreased. Figure 5 (1A) shows the reduction in heat transfer rate, as measured by the Stanton number, as the injection rate, as measured by the blowing parameter, is increased. The Stanton number (St) is defined by

St = q[peue(Ah)]

where $\dot{\mathbf{q}}$ is the heat flux; $\rho_{\mathbf{e}}$ is the density at the edge of the gas boundary layer; $u_{\mathbf{e}}$ is the velocity at the edge of the gas boundary layer; and Δh is the difference between the gas recovery enthalpy and gas enthalpy at the temperature of the wall. The injection rate as measured by the blowing parameter is the ratio of the mass injection rate to the product of the streamwise mass flux and the Stanton number without gas injection. Curves are shown for the injection of sir into air and hydrogen into air for both a

laminar stagnation point (3) and flat plate without gasphase reactions (4). In addition, an experimental curve is shown for air-air with turbulent flow on a flat plate (5). Thus (1) the heat transfer rate reduction is large in all cases, (11) hydrogen reduces the heat transfer rate more than air, and (111) turbulent boundary layer flow reduces the effectiveness of gas injection.

"The efficiency of an ablative material is frequently measured by the effective heat of ablation defined by

Heff # golm

where Heff is the effective heat of ablation, and qo is the heat transfer to a nonablating surface at temperature Ty. The effective heat of ablation includes gas-injection effects. The effective heat of ablation is strongly dependent on the exposure conditions as a result of the factors discussed above. For illustration, Fig. 6 (1B) shows the effective heat of ablation of ice as a function of the external temperature. The curves were computed at three pressures by Roberts (6) to show the effect of boiling point. The various components of effective heat of ablation are shown on the figure. It is clear that vaporization is the dominant factor and that the effectiveness of ice improves dramatically with increasing external temperature. At the leading edge of the vehicle, the external temperatures shown in Fig. 6 (1B) are equivalent to the stagnation temperatures shown in Fig. 1. The increase in effective heat of atlation with severity of environment is a desirable characteristic which is generally common to gas-producing ablators. To carry the illustration further, the previously mentioned flight condition of 6 km/ sec at an altitude of 30 km would lead to an effective heat of ablation in excess of 1200 calories per gram if the vehicle utilized ice as a heat shield."

Another method of application of this same principle could be achieved by selection of the proper plastic for the fabrication of the thermal protection system. In this case, a plastic is selected containing naphthene rings as an integral part of the molecular structure. For instance, such a plastic might be polymerized cyclohexylethylene, (i.e., hydrogenated polystyrene). Into this would be incorporated a finely divided or molecularly dispersed dehydrogenation catalyst. When this layer heated up the initial reaction would be to dehydrogenate the cyclohexyl rings giving a very effective ablative system, since as Ungar explains in the section quoted above, hydrogen is a very effective gas in reducing the heat transfer at the ablating surface.

An interesting comparison between the performance of various fuels has been made by Amin and Molder in a paper entitled Performance Comparison of Gun-Launched Scramjets for Various Fuels". The fuels compared are kerosene, triethyl aluminum (TFA), liquid hydrogen, and SHELLDYNE. The author used the following parameters for the scramjet engine powered gun-launched vehicle:

SCRUMET ENGINE PARAMETERS USED TO CALCULATE PERFORMANCE

Parameter	Value
Intake area ratio, A ₁ /A ₃ Intake efficiency, K _c ^a)	12.5 0.94
Ruel total pressure, psf Fuel total temperature, R	200,000
Downstream fuel injection angle, deg Combustor area ratio, As/As	145 3.5
Average pressure factor, 1.5) Exit/inlet area ratio, A7/A1	i

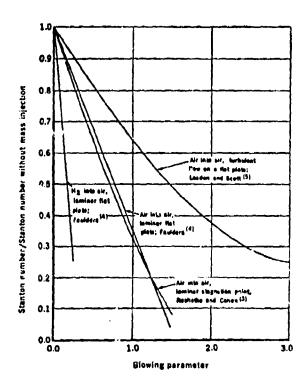
a) $K_d = \text{"process efficiency"} = (h_3 - h_1)/(h_3 - h_1)$ where the superscript prime denotes conditions obtained when the air at station 3 is expanded isentropically until $p_3 = p_1$ and h is static enthalpy.

b) Defined by p = p₃ + n(r₃ - p₅) where p is static pressure and p is the average pressure required to calculate the thrust on the combustor internal walls.

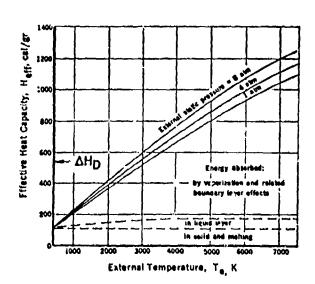
Although they evaluated both volume limited and weight limited vehicles, they consider the volume limited case to be more practical. The authors concluded that "SHELLDYNE was the best of the four fuels considered for volume limited scramjet vehicles. There is no significant theoretical performance difference between the TEA and kerosene but the former might give better performance in practice because of its greater reactivity. Nonoptimized single stage 90° gun-launched scramjet vehicles using these fuels are theoretically capable of accelerating a 200-lb payload from Mach 5 to Mach 9 during ascent and reaching an altitude of approximately 200 miles". For 90° launching, the actual values of the apogee calculated out to be 275, 215, 205 and 78 miles for SHELLDYNE, kerosene, TEA and LH2, respectively. At 45° launch, the range in miles (in the same order) would be 405, 316, 301, and 115.

In consideration of the state of knowledge with respect to the kinetics of hydrocarbon air supersonic combustion, it is apparent that there is a need for much further work in this field. This is evident from a study of the analysis of nonequilibrium hydrocarbon air combustion presented in the paper by W. Chinitz and P. Baurer⁷) which is a good summation of the complexity of the kinetic situation in this sort of system and the difficulty of agreeing on the nature and importance of such a fundamental parameter as the ignition delay time. In view of the problems which have been encountered in achieving successful piloting of a hydrocarbon scramjet especially if air rather than pure oxygen is used as the pilot flame oxidant, it is suggested that further investigation of the effect of various types of additives on the ignition delay and also on the oxidation rate following the onset of ignition is urgently needed. The first approach to obtaining this sort of data can probably best be made in a shock tube.

An interesting analysis of aircraft engines development was made recently by Λ . A. Lombard. He points out that the best way to increase the



A. Heat Transfer Reduction Due to Gas Injection



B. Variation of Effective Heat Capacity Heff of Ice With External Temperature Temperature

ΔHp = Heat of Dehydration of Cyclohexane

Figure 1. FACTORS AFFECTING ABLATIVE EFFECTIVENESS
Ungar, Science, Vol. 158, 1967

overall thermal efficiency of a power plant is to fly faster. A lot of difficult engineering in an engine is only necessary because of the low design speed - since the jet velocity must be low. Figure 2 is an illustration taken from Figure 52 in his paper which shows how the overall thermal efficiency of a range of engines varies with design Mach number. He states "this is an envelope curve in that from 0.8 to above 2 Mach number the curve represents the efficiencies of a range of turbo fan engines, from 2 to just below Mach 3 a range of pure jet engines and from Mach 3 onwards ramjets. It is very clear that at higher Mach numbers utilization of energy is very good indeed." And again, "as we go still higher in design Mach number the design of an airbreathing power plant becomes even more difficult. At a Mach number of 7 in the stratosphere the total temperature of the air entering the engine is over 2000°C and the combustion temperature must be of the order of 3000°C in order to achieve a worthwhile thrust. Quite apart from the metallurgical problems, there are big problems connected with the dissociation of the products of combustion of normal fuels which makes the attainment of these temperatures difficult."

It is evident that considerable incentive exists for the development of a multiple purpose additive. One which will act as a dehydrogenation catalyst in the heat exchanger of the engine, will act as a combustion catalyst in the combustion chamber and will act as a recombination catalyst in the nozzle section of the engine. Until it may be visionary to hope that this can be relieved, there are compounds of metals and oxides that have properties which encourage us to continue dreaming.

Some interesting papers having to do with heat transfer and boundary layer flow have appeared recently which are pertinent to the present investigation. Levis, Kubota and Lees published an article in the A.T.A.A. Journal for January 1968, p. 7 on an "Experimental Investigation of Supersonic Laminar Two Dimensional Boundary Layer Separation in a Compression Corner With and Without Cooling". They compared the surface pressure distribution for cold wall with the adiabatic configuration for a Laminar interaction and observed the dependence on Reynolds number for both laminar and transitional interaction. The free interaction similarity suggested by Chapman was empirically tested and found to be a good approximation for the adiabatic configuration but it failed to correlate the cooled case with the adiabatic case. The scaling suggested by Curle was tested and found to eliminate this deficiency.

Also in the same journal, p. 15, the paper by Barry E. Edney¹⁰) related the "Effects of Shock Impingement on the Heat Transfer Around Blunt Bodies". He reported that an extraneous shock impingeing on a blunt body in hypersonic flow altered the flow around the body and increased the local heat transfer rate near the impingement point. A physical model was set up which predicted variations in shock interference patterns and surface pressure distributions and of the intensity in the extent of the peak heating, in accordance with the experimental findings.

The February issue of the same journal (p. 193), Laganelli, Aires, and Hartnett¹⁾ wrote on the "Transpiration Coolin, in a Laminar Boundary Layer Solid Wall Upstream Effects" in which they developed an analytical model for this type of cooling. It was found that the solid leading edge had a significant effect on the magnitude of the skin friction coefficient over the transpiration surface. They concluded that further investigations would be

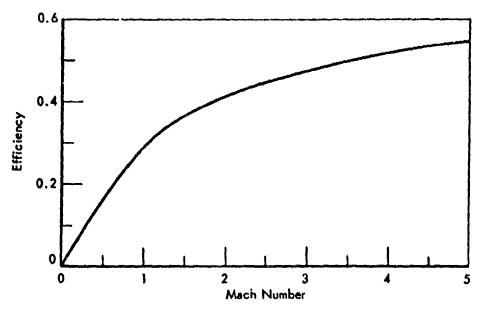


Figure 2. VARIATION OF OVERALL THERMAL EFFICIENCY WITH MACH NUMBER

required for transpiration mystems particularly systems which include a non-porous leading edge, since this increases local skin friction.

Also, F. W. Spaid and E. E. Zudukoski¹² describe a "Study of the Interaction of Gaseous Jets From Transfer Slots With Supersonic External Flows". This was an analytic and experimental investigation at Mach numbers up to 4.5. A correlation of data obtained from experiments with finite span slots demonstrates that the effect of jet penetration height and the slot span are the important characteristic dimensions of such flow fields.

In the March issue of the A.I.A.A. Journal, E. J. Felderman¹³⁾ on p. 408 writes on the "Heat Transfer and Shear Stress of the Shock Induced Unsteady Boundary Layer on a Flat Plate" in which he examined the development of the boundary layer following the passage of the initial shock wave over a semi-infinite flat plate mounted in the shock tube. Both experimental and analytical solutions of the problem were sought. Data obtained over a range of shock Mach numbers, initial channel pressures and position on the plate agreed well with the theoretical calculations.

An interesting paper on the "Diffusion of Gases and Porous Solids Over a Thousand-Fold Pressure Renge" was offered in Chemical Engineering Science 1957 Vol. 22, p. 11 by Henry Cunningham and Jim Geankoplis¹⁴ in which they showed that the experimental diffusivities of gases in alumina and in porous Vicor compared closely with those predicted by the raw field effectiveness factor model and the Wakao and Smith random pore model, as did the data on Vicor.

A paper by M. F. L. Johnson and J. Modiss in the Division of Petroleum Chemistry Preprints, ACS Chicago Meeting, September 1967 on the origin and types of pores in some alumina catalysts andicated that the authors were able to distinguish as many as three different pore systems in clumina and alumina catalysts. This data is mainly useful in calculating effective diffusion coefficients.

A paper by S. Landa, J. Vais, and J. Berkhard published in the Collection Czech. Chem. Commun. 32 No. 2, p. 570, February 1967 dealt with adamatene and its derivatives. This is interesting because of the possibility of using an adamantane derivative as a high density thermally stable fuel.

A paper by Kumugi, Tominaga, and Abikozi) on a kinetic study of the pyrolysis of propane in the presence of hydrogen appeared in the Sekieyu Cakkai Shi, No. 11, 1966 (Japan).

An article appearing in Kinetic Catalysis Chemical Engineering Symposium Series No. 73 Vol. 63, 1967 by R. L. Smith and C. D. Prater on "Some Capabilities and Limitations of Kinetic Studies in Heterogeneous Catalysis as Illustrated by Cyclohexane-Cyclohexane-Benzene Inter-Conversion Over A Supported Platinum Catalyst" shed some light on the mechanism of this important reaction. They gave some kinetic information on the various steps involved with the general conclusion that cyclohexane is not a necessary intermediate in the dehydro_enation of cyclohexane.

Laboratory Reaction Studies

Laboratory studies of candidate endothermic fuels and catalyst systems were continued in our bench-scale reactors.

The relative reactivities of SHELLDYNE, hydrogen treated SHELLDYNE (SHELLDYNE H) and Decalin for thermal reaction were determined.

A pulse reactor was constructed for use in studying catalytic reactions under fixed bed and dispersed phase conditions. A brief study of the dehydrogenation of methylcyclohexane over our standard 1% Pt on Al₂O₃ catalyst was done in this system at liquid hourly space velocities (IESV) up to 4265.

Several commercial and laboratory-developed catalysts that were promising for the dehydrogenation of methylcyclohexane were evaluated for the dehydrogenation of Decalin in our conventional flow reactor.

Studies on the dehydrogenation of Decalin, tetralin and mixtures of the same were carried out in a highly diluted catalyst bed, in order to obtain kinetic data for formulating a mathematical model for this reaction.

A few catalysts that appeared promising for the dehydrogenation of methylcyclohexane under micro-scale testing (MICTR) were further evaluated in our bench-scale reactor.

Thermal Reaction of SHELLDYNE H and SHELLDYNE

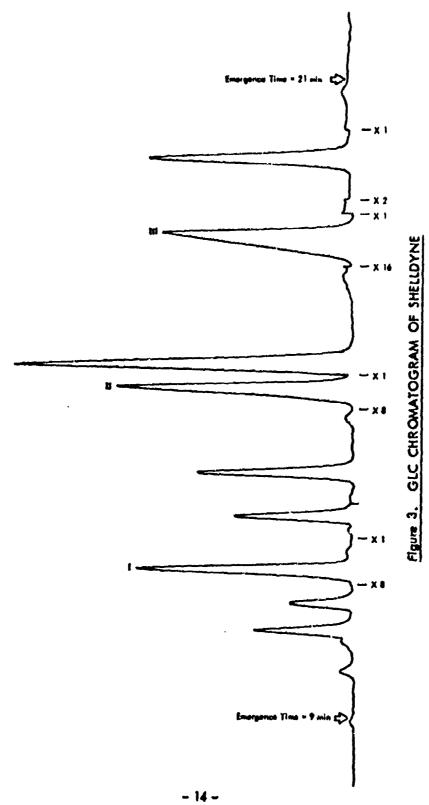
SHELLDYNE is the trade name of a high density, high energy fuel developed by Shell³) for use in air breathing and rocket engines. This fuel is not prepared from crude oil by conventional processes, but is manufactured from specific chemical intermediates. It has moderately good storage and thermal stability which is improved by hydrogen treatment. As SHELLDYNE is being considered for use as an advanced fuel both SHELLDYNE and hydrogen treate's SHELLDYNE (SHELLDYNE H) were evaluated for thermal reactivity in our bench-scale reactor.

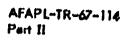
Some physical properties of SHELLDTNE and SHELLDTNE H are tabulated below. It is evident that hydrogen treatment increased the viscosity slightly and somewhat lowered the density, RI, freezing point and heat of combustion (Table 1).

SHELLDINE consists of numerous isomeric compound of which three isomers made up 85% of the material. These were present in the ratios: I:II:III = 16:21:48. Figures 3 and 4 show GIC analyses of SHELLDINE and SHELLDINE H respectively. The chromatographs have the same general pattern, but the emergence times of the SHELLDINE H components are longer however.

Both SHELLDYNE and SHELLDYNE H were tested under conditions of vapor phase thermal reaction at furnace block temperatures of 752-1202°F and 1-10 atm pressure. Both feeds were tested in a 1/4" CD stainless steel tube

a) Information and experimental quantities may be obtained from Shell Oil Company, Products Application Department, New York.





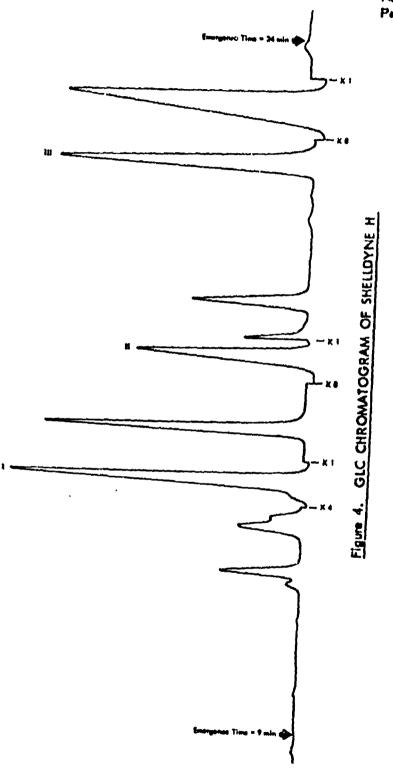


Table 1. SOME PHYSICAL PROPPRTIES OF "SHFLIDYNE" AND "SHFLIDYNE" H

	SHELLDYNE	SHELLDINE H
Boiling Point	482-89	500
Freesing Point, "F	-24	(-85) a)
Specific Gravity, 60°F/60°F	1.100	1.081
Viscosity, os 212°F 68°F	2.89 2.89	5.19 26.2
Refractive Index (68°F)	1.5479	1.5401
Pounds/gal (US)	9.16	9.02
Hear of Combcetion, Btu/gal	163,830	160,590

a) This material did not show a sharp freezing point but became more viscous with decreasing temperature. At -85°F the liquid could no longer be stirred.

with no packing in the tube. In addition SHELLDINE was tested in our 5/8" ID standard reactor tube packed with 10-20 meah quarts chips.

In the 1/4" OD tube the wall temperature was measured at seven points along the tube. The points were 1-1/2 inches apart and the top point was one inch below the top of the secondary liner (Figure 5). The portion of the tube above the secondary furnace liner served as a pre-heat section and was kept at TTO*F. The temperature of the reactor wall varied down the tube and Figure 6 shows the temperature variation for a furnace block temperature of 1202*F.

The maximum reaction rate will occur in the region of maximum temperature. Presumably the rate in that portion of the tube whose temperature was 18°F (10°C) or more below the maximum temperature, did not contribute appreciably to the overall rate. Thus the "effective" volume of the tube was that portion of the tube whose temperature was within 18°F of the maximum well temperature, and whose volume was determined from a plot such as Figure 6. The "effective" reactor temperature was taken as 9°F below the maximum temperature and space velocities and Apparent Contact Times (ACT) were calculated based on the effective volume and effective reactor temperature.

In the 5/8" ID reactor tube the space velocities and contact times were based on the volume of quartz chips (20 ml) and the reactor wall temperature as measured by a thermocouple pressed between the reactor well and the furnace block.

a) This reactor system was described in a previous report in detail, on p. 248, 254.1)

Liquid product material was analyzed by GIC using a 165° capillary column (0.018" diam) coated with 20% polyphenyl ethers in DC 710 silicone. Gas products were analyzed by mass spectrometry. Conversions were calculated from product analyses and neglect coke or polymer formed during reaction.

SHELLDYNE H

At short contact times (0.3 to 0.8 seconds) SHELLDYNE H was reasonably stable. Thus conversions were less than 3% at 1022°F and only 5 to 10% at 1202°F (Table 2). At these contact times conversions were generally lower at higher pressures probably because the increased mass flow at the higher pressures reduced the reactor temperature. At longer contact times (3-4 seconds) increased conversion was observed. Highest conversion obtained was 51% (1202°F, 10 atm) of which about 8% was to light has products. The liquid product was black, but no tube plugging occurred. Product material was principally cracked liquids (i.e., lighter than starting material) with some light gas at the higher conversions (Table 5). The three isomers were not equally reactive and in general at 1202°F, I was the most reactive at lower pressures and III at higher pressure (Table 5).

First order rate constants were calculated from the rate of disappearance of starting material. Using these values activation energies of 41.8 and 40.3 kcal/mole were obtained over the temperature range of 1022 to 1202°F (Runs 110, 112-1 and 111, 112-3; Table 2).

SHELLDYNE

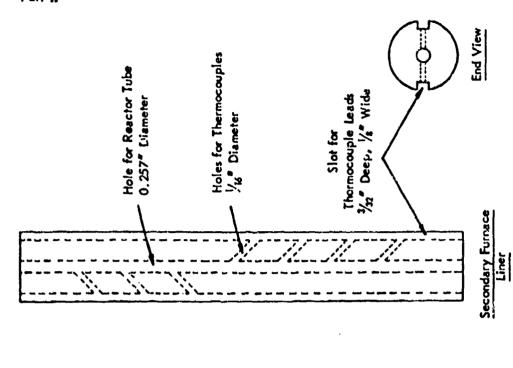
SHELLDYNE was more reactive and less stable than the hydrogen treated material (Table 4). Thus in the 1/4" On tube conversion at 797°F (Run 114) was about that obtained with SHELLDYNE H at 1202°F! (Run 112-3; Table 2.) Quantitatively, based on the first order rate constants and an activation energy of 41 kcal/mole, SHELLDYNE was 1500 times more reactive than SHELLDYNE H. Further, at 797°F considerable coking occurred with SHELLDYNE and the reactor began to plug after 15 minutes (Run 115, Table 4). As with SHELLDYNE H the various isomers were not equally reactive and generally the reactivity of I = III > II (Table 5). At constant contact time, pressure did not appear to affect reactivity (of Runs 114 and 115 Table 4).

In the 5/8" ID reactor tube at higher temperatures and longer contact times coking was even more severe. For example at 932°F (3.6 seconds ACT) the reactor plugged after 15 minutes while at 1022°F (4.5 seconds ACT) plugging occurred after only about five minutes reaction time! Further, before the tube plugged a hot spot appeared and moved down the quartz bed and the temperature in the bid rose to over 1292°F even though the block temperature was only 1022°F!

In both sets of experiments the reaction products were principally liquid cracked material and light gas.

Comparison With Decalin

In order to relate the stability and reactivity of SHELLDYNE H and SHELLDYNE to the naphthenes examined earlier, 1)3) a few experiments were done with F-ll5 Decalin in the 1/4" OD reactor tube. The composition of



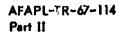


Reactor Tube

Secondary Liner

Mechanite Furnace Liner Figure 5. SECONDARY FURNACE LINER FOR 1/4" Q.D. REACTOR TUBE

Heating Elements



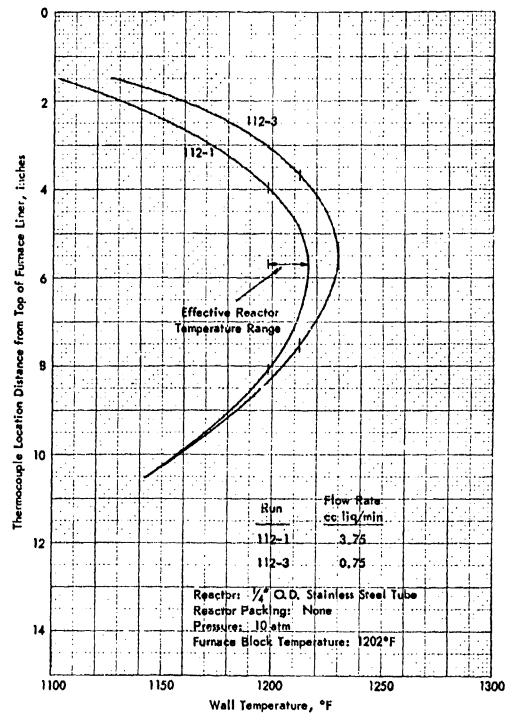


Figure 6. REACTOR TEMPERATURE PROFILE

Table 2. THERMAL REACTION OF "SHELLDYNE" H

Reactor: 1/4" CD Stainless Steel Tube Reactor Packing: None

								T		1	ļ							
Pam 105k8-	98	ጷ	8	101	1-201	102-2	103	701	žõ	28	5	1.8-1	100-2	8	110	111	112-1	2-211
Pressure, atm	1	5		5	10	7	5	2	7	~	2	[Ĭ,	å		° I		
Feed Flow Bate, ml/min	0.75	×.73	6.73	3.73	3.	6:3	3.73	8.	0.75	3.75	8.	0.75	3.75	8.	3.73	12:0	3.75	0.75
Temperature, 'F																		
Mock			150		T		1 28		Î	1022 -	1		12.22	•	1022	R	1200	2
Vali	797	781	3	<u>8</u> 6	Ę	835	819	812	*	975	927	1155	11.86	30%	8	953	1103	211
	88	į.	8	ě	8 2	3	8%	35	1024	1006	253	1202	1177	8	200	1322	यग	1197
	88	108	808	8	ê	855	878	83	9	1026	101.5	7556	120	1191	10.26	9	1195	1209
	88	58	8	8	160	655	\$	637	0401	1031	10.2	1221	1216	1302	1031	100	Ę	1233
	8	108	8	8	762	20	3	3	1028	7020	1017	र्ग व	1306	**	1021	8	7500	\$171
	82	26	8	82	33	857	87,8	8	7001	2001	8	181	1182	1175	300	1010	1180	1182
	#	Ė	٤	Ė	Ę	815	Z,	815	\$7.6	3	426	311	1111	1161	156	#	1143	1143
Effective Reactor Temperature, 'F	757	79.5	\$2	393	82	35	8	83	1031	1022	1013	0221	ដ	1195	2001	zz oz	1,306	827
Effective Reactor Volume, al	3.36	3.6	3.37	40.5	3.51	\$	89.	2.7	1.95	27:-1	8:	1.64	1.54	8	1.93	2.13		1.7
Effective LHSV	13	2	13	2	3	2	87	\$	53	11.7	ğ	۶	Ã	8	Lit	`	297	ж
Effective ACT, sec	0.80	0.70	0.80	٠ 5	8.0	4.0	8	39.0	%	8	я 6	0.8	0.27	9.38	0.75	2.3	61.0	3.0
Stelland Conversion, &	3.1	9.6	0.3	0.3	1.8	0.3	1.6	9.0	2.6	9.0	2.4	2.01	4.9	5.3	8.8	5.6	25.1	51.0
Mrst Order Rate Constant, sec"3	•	'	,	•	,	'	,	,	-	•	•	0.372	0.346	0.208	0.039	0.91k	0.59	0.236

Table 3. THERMAL REACTION OF "SHELLDYNE" H

Product Analyses

Reactor: 1/4" OD Sisirless Steri Tube
Reactor Packing: None
U, = emerged before I, includes cracked liquid
U2 = emerged after I and before II
U3 = emerged after II and before II
U4 = emerged after II

Run 10548-	Foed	8	8	8	101	102-1	102-5	ő	ğ	502	8	101	1.98-1	108-3	<u>8</u>	617	ä	112-1	112-5
Pressure, stm		1	\$	-	s	ន្ន	-	~	o.	=	1	2	-	^	9		۱		
Temperature, "F																	-		
Lock				121				- - }		-	- 7025	1	-	1,202,1		- 8		- 22	1
Effective Mall				5	ŝ	82	3	3	3	1631	1022	1013	23	ızı	1195	2201	1031	9021	1230
Product Amalysis, &							-												
ដ	2.1	2.6	2.0	2.8	8.5	8.8	5.9	2.8	5.9	3.0	0.	7.	10.1	6.3	5.6	*	7.0	3.61	
	15.5	15.2	14.7	14.9	14.6	14.5	24.9	19.7	14.9	13.5	14.8	14.4	13.0	12.9	P. 8	14.6	7.41	13.0	7.6
*3	2.8	2.8	89.	2.1	2.8	5.1	5.5	9.9	8.	?	2.9	2.1	3.6	3.3	1.	2.9	3.0	3:3	5.5
11	8.2	23.2	\$.5	2.2	22.7	2.0	25.1	23.1	23.1	21.6	8.8	25.2	8.5	21.9	21.5	Š	8.8	17.71	12.5
s n	. .	3.6	3.8	7.4	1:	9:	3.8	1.4	5.1	\$.0	8.6	4.7	2.4	÷	9.	19	5.6	5.5	3.8
111	20.00	57.5	2.2	3	Ŕ	\$1.8	51.5	51.9	51.9	9.8	51.9	51.3	7.7	<u></u>	1.8.1	8	47.3	3	.8.0
*	0.7	9,	1.2	1.2	0:	0.1	6.0	6.0	1.1	1.0	0.1	1.6	1.2	8.0	9.0	1.6	9.1	2.1	9.
Light Ges	•		•	,	•	•	•	•	•	•	,	•	•			,	,	4.3	10.8
Conversion, &				_							-	•							
H	•	2.0	~	0.4	5.9	6.5	0.	5.2	0.4	15.0	9.	1.2	16.2	17.8	1.6	o,	5.2	16.2	4:4
11	•	3	4:1	6.5	6.0	3.6	7.7	3.2	6.5	5.4		2.1	11.5	0	6.8	0:1	8.8	2.4	
111	,	2.2	رن دن	2.5	4.0	1.6	2.5	1.4	1:	0.0	4.	5.6	4.6	9.2	8.6	4.5	1.01	 8: 1	6:39
Total	•	7.0	9.0	6.3	6.5	1.8	0.3	1.6	9.0	5.6	0	4.0	10.2	6.7	5.3	9,	5.6	25.1	51.0

Table &. THURMAL REACTION OF "SHELLDYNE"

Rum 10548-	36	56	8	111	115	79	59	19	89	2	1-02	Ř
Reactor	% 1→	38 srt	Inless	at a	← 1/4" OD stufnieus eten! tube →	V	5/8	5/8" 19 eteinless steel tube	2 m le	<u>is</u>	 4	$ \uparrow$
Resctor Packing			3		1	V	5	- Oceantz chips; 10-20 mesh-	-01 :38	8		1
Reactor Volume									23			1
Pressure, ata		S	2	-	v,	, 2	-	_		-	3	12.
Food Flow Rate,	0.75	0.75	0.75	0.75	3.75							\uparrow
Tesperature, T			_									
Block			- 787 -		$\bar{\uparrow}$	2201	25	727	842	832	932	332
	778	8	8	52	E	935	716	Ē	83	8	8.2	6 38
	<u>\$</u>	799	8	8	8	•	•	•	,	•	•	•
	88	ğ	2	ž	28	•	•	•	•	•	•	•
	88	8	810	8	799	•	•	•	•	•	•	•
	203	802	8	æ	799	•	•	1	•	•	,	•
•••	782	262	386	787	25	•	•	•	٠	•	•	•
	222	114	E	772	Ĕ	•	•	•	•	•	•	•
Effective Resotor Temperature, F	797	797	8	787	8	•	•	•	•	•	•	•
Effective Reactor Volume, al		7	3.1	3,0	3.4	•	•	;	•	•	٠	•
Effective LHSY	±.5	14.5	14.5	51	99	V			F			1
Effective ACT, seconds	0.7	3.6	7.1	0.63	62.0	87.	0.55	0.54	81.0 12.0	0.48	2.[3.6
\$ SHELLDTHE Converted to Light Eas	9.7	ង	1.0	5.7	3.0	•	<u>.</u>	(q. 5	80	9.4	7.0	5.4 35.4
SKELLDTAE Conversion, Se	۶) درو	62.9	79.0	24.0	(p3°52	()(3°88	3	3.6 (1)	8.61	17.7	83.8	90.0 ^d)
First Order Rate Constants, exc-1	26.490	0.490 0.274 0.216 0.391	0.216		0.382	•	•	•	·	٠	•	•
A Mariante and a family of												

a) Majects conversion to light gas.
 b) First rate not steady during the run. Conversion maginets light gas fraction.
 c) Reseter plugged after "5 minutes.
 d) Reseter plugged after "5 minutes.

Table 5. THERMAL REACTION OF "SHELLDINE"

Product Analyses

U₁ = energed before I, includes cracked inquid U₂ = emerged after I and before II U₃ = emerged after II and before III U₄ = energed after III

70-3		_ 1:	9.50 9.63	0.0	4.4.4 4.4.6	35.4	1004)	26.86 9.86	8.0.5
70-1	eqn	£ 8	55.8	8.4. 8.4.	1,5 4,6 4,6	9.9	74.0	27.0 83.8	63.8
8	teel t		41.2	0.0 8.0	5. V.	9.40	53.4	4.4. 8.8.	47.7
88	stainless ste Quarts chips	842	13.1	4.0	0 0 0 1 0 1 0	0.4 0.4	28.8	,3 % 5.25 5.45 5.45 5.45 5.45 5.45 5.45 5.45	19.8
L9	5/8" ID stainless steel tube	32	0.4	4. c.	18.2 5.3.2 5.3.2	5.0 9.2b)	8.38)	13.38	3.6
<u>ک</u> ن	5/8"	82,5	2.8	15.2	8 w क v v &	5.3	4	01 ± 1	5°C
ಪ		1022	5.2	0.0	0.5 5.5	5.0 (e)(e)	100,	5.85 6.65 6.65 6.65 6.65 6.65 6.65 6.65	
115	**************************************	1					ر ا ا	26.9	25.0
114	eel tu	1	18.6	4.0	25.25. 25.24.	0.V	24.0	200	U. #>
96	(-1/4" OD stainless steel tube ->	797	4.0	9.0	× + 1 %	10.4	87.0	989	0.6)
93	stainles	101	28.2	0.0	12.5 13.5	5.7 33.1	45.8	\$ C.3	8.3
76	9 -		19.8	000	5 v.V.	± 6.	4.72	3.7.5. 5.4.5.	27.0
Feed	1		2	2.9	1.00	0.0		1 1	•
Run 10548-	Reactor Reactor Packing Reactor Volume Pressure, atm	Temperature, F Block Fffeoffwe uxil	Product final., Sw	is n	i si i	U. Light Cas	Conversion, pr	III	TONGT

a) Neglects light gas.
b) Values suspect due to flow rate not steady during run.
c) Reactor plugged after about 5 minutes.
d) Reactor plugged after 15 minutes.

this Decalin (DHN) was 25.1% trans-DHN, 74.5% cis-DHN and 0.4% tetralin. The data for a series of runs at 1202°F, 10 atm pressure, and various contact times are presented in Table 6. With this naphthene no coking was observed, although the product at the highest contact times was dark (Run 120). Comparing conversions and rate constants for Runs 119-3 and 120 (Decalin) with Runs 112-1 and 112-3 (SHELLDYNE H; Table 4, values in parentheses Table 6) it appears that SHELLDYNE H is slightly more reactive than Decalin.

Discussion

From the work completed thus far it is evident that SHELLDYNE is highly reactive when in contact with metal at 797°F and higher. Hydrogen treatment improves stability and reduces reactivity by a factor of about 1500. Based on conversions comparable reactivities for SHELLDYNE H were observed at temperatures 550-400°F higher than for SHELLDYNE. Pertinent data are summarized in Table 7.

Pressure did not affect the reactivity of either species. Hydrogen treating increased the viscowity by about 10-30% and decreased the density by about 2%.

Presumably the metal reactor catalyses the decompositions of SHELLDINE to coke as other researchers have pyrolysed SHELLDINE components in a Pyrex tube cleanly and without coking at temperatures up to 642°F and at contact times up to 6 minutes. Some studies on the effect of metal surface on SHELLDINE reactivity will be done.

From the work carried cut thus far it is evident that at elevated temperatures SHELLDYNE H has much greater stability than SHELLDYNE, and hence is a more attractive high density fuel under high temperature conditions. In a later section of this report (page) it is shown that in Coker-type thermal stability tests SHELLDYNE H was also more stable. Thus it appears that SHELLDYNE H was superior to SHELLDYNE on all counts except for a 2% loss in gravity and heat of combustion and a small increase in viscosity (Table 1).

Dehydrogenation of Methyleyelohexane in a Pulse Reactor

A small pulse reactor has been constructed for studying catelytic reactions under both fired bed and dispersed phase conditions. Some of the advantages of such a system over the conventional flow reactors are:

- a) Heat transfer effects within the reactor can be virtually eliminated.
- b) It is a rapid method for studying reactions under varying conditions.
- c) High space velocities can be achieved with only a small amount of feed.

To date the pulse reactor technique has been confined primarily to exploratory and reaction mechanism studies. Recently, however, R. P. Merrill²⁰) has suggested a method for obtaining reaction rate data from pulse reactor studies.

Table 6. THERMAL REACTION OF DECALIN

Reactor:

1/4" 00 Tube

Reactor Packing:

None Block Temperature: 1202°7

Feed:

F-113 Decalin

Run	117	118	119-1	1119-3	120
Pressure, atm	1	5	10	10	10
Feed Flow Rate	0.75	3.75	7.50	3.75	0.75
Wall Temperature, *F	1157 1202	1132 1185	1094 1166	1128	1150
	1227 1227 1211	1215 1211 1202	1197 1195 1193	1211 1211	1222 1208
	1184 1143	1119 1144	1172	1179 1143	1182 1143
Effective Reactor Temperature, *F	1218	1204	1188	1202	1213
Effective Reactor Volume, ml	1.46	1.42	1.75	1.42	1.36
Effective LHSV	31	158	257	158	33
Effective Contact Time, sec	0.25	0.25	0.31	0.50	2.36
DHN Conversion, %w	3.0 (10.2)	4.8 (7.9)	2.1 (5.5)	13.9 (25.1)	59.6 (51.0)
First Order Rate Constant, sec	0.136 (0.372)	0.218 (0.346)	0.079 (0.208)	0.517 (0.594)	0.405 (0.236)

TRULE 7. COMPARISON OF REACTIVITIES OF "SHELLDINE" H AND "SHELLDINE"

	ELLDYNE H	<u> </u>		HELLDYNE	
Block Temperature,	Contact Time, Sec	Conversion,	Block Temperature,	Contact Time, sec	Conversion,
1505 1055 1055	0.8 4.2 3.0	2.8 5.6 51. 0	752 797 797	0.5 0.7 3.6	5.0 24.0 51.0

Talkett and seems and an expension

A brief study was made of the dehydrogenation of methyloyoloherane reaction using the pulse reactor technique. In this system a stream of carrier gas flowed through the reactor continously. At the desired time a small amount of feed (ca I microliter) was injected into the carrier gas stream and subsequently passed over the catalyst as a "pulse". Reaction products or a slip-stream sample thereof, were led directly into a GLC for analysis. In the pulse reactor system there was virtually no heat transfer affect within the reactor. The apparatus and procedure for carrying out the experiments are described in detail in the appendix.

Experiments were carried out at 10 atm pressure, 572° to 752°F and at carrier gas flow rates that correspond to MCH liquid hourly space velocities (IMSV) up to 4265. Both helium and hydrogen were used as carrier gas; one microliter of liquid MCH was injected as a pulse. The catalyst was 0.25 g of our standard 1% Pt on Al₂O₃ laboratory catalyst and was diluted with 1.25 ml quarts chips to give a total bed length of 3 inches.

In this resutor system high MCH conversions were obtained at much higher space velocities and much lower reactor temperatures than were obtained in the bench-scale test reactor. For example, 95% MCH conversion was obtained in the bench-scale reactor at 1200°F and IHSV of 100, and in the pulse reactor at 662°F and IHSV of 1000. Further, at 752°F in the pulse reactor 85% MCH conversion was obtained at an IHSV of \$265.

Conversion declined with increased space velocity, as shown in a series of bracketted runs at 662°F with helium carrier gas (Table 8).

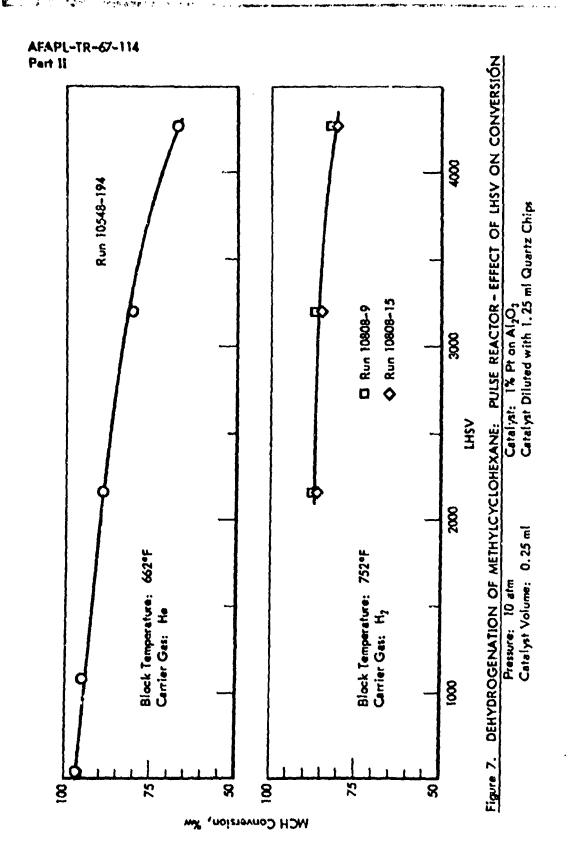
Figure 7 shows conversion as a function of IRSV. At the highest IRSV tested (4265), an MCH conversion of 68% was observed. Selectivities for toluene were 99+%. Catalyst activity did not change during these tests as the conversions were about the same in the initial and final runs.

At higher temperature there was cutalyst deactivation in a series of tests at high space velocity. Thus at 752°F at LESV of 4265 with helium carrier gas, conversion declined for 71% to 60% with successive MCH pulses (Table 9; Runs 5, 6-1, 6-2).

Injecting larger MCH pulses (10 ml) seemed to enhance the deactivation (Table 9, Runs 6-3, 7-1, 7-2). Changing to H₂ carrier gas appeared to restore activity somewhat (Table 9; Runs 8-1, 8-2).

Table 8. DENTOROGENATION OF METHYLCYCLOHEXANE: PULSE REACTOR

Run No. 10548-	104	135.1	6-70	18.	1. 4.	<u>7</u>	106.9	106.3	1,401
UESV		15.38	が	2150	が	288	N A	また 5524 年5 0025 年5 0512 本5 8C71 本5	1 20
Temperature, 'F	Ke1, cg	73-047	83 637	4	20.00	Ç	,	į	3
Preheater	70°-10°	55.44 5.45	748	79.	689 89	8 8 8	119 oc-160	56-150 56-150 56-150 56-350 56-350 56-150 56	051-0 147
Product Analysis, &									
Cracked, 11q	1.8		9.0			6.9	0.5		Ö
HCH	3.6		2.7	10.8		18.7	2.3		
Toluene	9. \$	きる	7.98		98.8	81.0	7.%	67.8	. % 4:
MCH Conversion, for .	%	8.	97.3	97.3 89.2	97.3	81.3	97.2		%
Selectivity for Toluene, for	98.2	4.66	4.66 5.66	*. 86	8.5				



Teble 9. Desidence watton of methylotochekane: fulse reactor

Cetalyst Stebility

Cetalyst: 15 Pt on Al₂O₃ Pulse Volume: 1 14, ilquid Cetalyst Volume: 0.25 ml Pressure: 10 etm Peed: Pure MCH Temperature: 752°F Cetalyst Diluted With 1.25 ml Quarts Chipo

Rm No. 10808-	5	1-9	2-9	6-38)	5 6-1 6-2 6-3*) 7-1*) 7-2*) 8-1 8-2	7-28)	8-1	8-2
Carrier Gas	V			8		Î	← He →	Î
LKSV	1,265	3200	4265	42654)	4265	42654	4265	4265 4265
Temperature, 'F								
Vell	745-48	743-48	743-48 743-48 743-48 743-48	743-48	743-48	743-48	743-48 743-48	743-48
Preheater	84.2	8	869	842	840	842	85 85	83.2
Product Analysis, %			•					
Cracked, 11q	3.4	2.1	6.0	0.5	9.5	0.2	0.3	0.8
MOR	28.9	29.9		•	世法	59.0	35.8	32.1
Toluene	67.7	68.0	59.3	47.9	45.1	£0.8	6.5	67.7
MCR Conversion, %	71.1	70.1	8.9	48.34	45.64)	41.14)	<u>\$</u>	6.79
Selectivity for Toluene, &	% %	97.0	8.3	99:1	99.0	8.3	99.5	7.66
s) 10 uf MCH injected before this pulse.	e this p	ulse.						

Higher conversions and no catalyst deactivation were observed at 752°F using hydrogen carrier gas. Thus at LHSV of 4265, 83-85% MCH conversion was obtained, and in a series of bracketted runs the initial and final conversions were about the same (<u>Table 10</u>). Selectivities for toluene were 92+% and more cracked products were made at the higher temperature. Conversion as a function of space velocity is shown in <u>Figure 7</u>.

Table 10. DEHYDROGENATION OF METHYLCYCLOHEXANE: PULSE REACTOR

Effect of Space Velocity on Conversion at 752°F

Catalyst: 1% Pt on Al₂O₃ Pressure: 10 atm Catalyst Volume: 0.25 ml Carrier Gas: H₂ Feed: Pure MCH Temperature: 752°F Pulse Volume: 1 µ\$, Liquid Rum 10808-9 Catalyst Diluted With 1.25 ml Quartz Chipe

Run No. 10808-	9-1	10-1	10-2	10-3	10-4	11-1
VEHJ	4265	4265	5200	4265	2150	4265
Temperature, °F						
Well	743-47	743-47	743-47	743-47	743-47	741-47
Preheater	882	882	959	871	874	851
Product Analysis, 4w						
Cracked, liq	2.6	6.6	5.9	5.0	6.5	5.9
MCH	17.6	15.5	13.2	16.0	11.9	17.2
Toluene	79.8	77.9	80.9	79.0	81.5	78.9
MCH Conversion, w	82.4	84.5	86.8	84.0	88.1	85.8
Selectivity for Toluene, #w	96.9	92.2	93.2	94.0	92.6	95.2

The activity of freshly reduced catalyst was considerably greater in the pulse reactor than in the bench-scale reactor. This could have been because in the latter system the catalyst was pretreated with pure MCH for 10 minutes prior to starting the runs and hence partially deactivated. Accordingly a series of experiments was done in the pulse reactor after pretreating the fresh catalyst with MCH. The pretreatment consisted of flowing pure MCH over the fresh catalyst for 15 minutes at 10 atm pressure and 662°F (no H2 or H2 present). The reactor was then flushed with helium and successive pulses of MCH were injected over the catalyst. For tests at 752°F, H2 was the carrier gas; at lower temperatures He was the carrier gas. The data are tabulated in Table 11 in the order in which the experiments were done. The conversion values obtained with no catalyst pretreatment are shown in perentheses for comparison.

Table 11. Desidence dution of methylcyclohexane: fulse reactor

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いかが、100mmの日本のの「中央の人」というできない。 では、100mmの日本ののでは、100mmの日本ののでは、100mmの日本のでは、100mm

Priect of Catalyst Pretreatment on Stability

Catalyst pretreated with pure MCH at IHSV of 100, 10 atm press., and 662°F for 15 min prior to these runs. Prise Volume: 1 µf, Liquid prior to Pressure: 10 atm Catalyst Diluted With 1.25 El Quartz Chips 1¢ Pt on Al₂O₃ 0.25 ml Pure MCH Catalyst: Catalyst Volume: Feed:

길 1	2150	702-64	8.4.3 84.3 85.3 85.3	
14-1 14-2 14-3 15-1 15-2 15-3 15-4 16-1 16-2 16-3 17-1 17-2 17-3 17-4	4265	652 662 	25.0 25.0 74.1 75.0 88.8	
221	3700 4265		6.1 8.8 8.08 8.5 8.5 8.8 8.8 8.8 8.8 8.8 8.8 8.8 8.	
12.	2150	396-702	1.6 13.7 86.3 1.86.3	
<u></u>	<u>نڊ ا</u> ر	662 653-57 6	3.58 3.58 8.58 8.58	
2-9	1068	662 655–58	•	P.
<u>-</u>	4 365	653-57 696-702 696-702 743-47 743-47 743-45 745-47 806 802 851 914 903 963 932	4.2 18.2 77.6 81.8 (82.8)	95et 94e2 97e8 54e5
ᆁ	1200 2150 4265	743-45 963	6.3 12.6 81.1 87.4 (88.1)	95. 9.
〗	320C 3	752 743-47 743-45 914 903 963	5.0 14.6 80.4 85.4 (86.8)	24.2
<u>2</u>	4265	31.6	3.7 5.0 19.5 14.6 76.8 80.4 80.5 85.4 (82.4) (86.8)	
ᆁ	21.50	707 36-702 851	0.88 0.88.0 89.8	0.88 0.88
<u> </u>	1068	707 696-702 6	0.8 4.t 95.1 95.8	98.2
2	1008	662 653-57 6 806	0.2 4.4 95.4 95.6 (95.0)	8,68
<u>=</u>	£ 5	662 533-57	7°0 6.88 88.88 7°89 8°4 8°5)	8°65 9°65
1				J.
-808). «M ← .0	Carrier Gas UKSY	Issperature, °F Furnace Vall Preheater	Product Analysis, Se Cracked, liq MCH Tolumno MCH Conversion, Se	Selectivity for Toluene,

. .

At 662°F conversions with the MCH pretreated catalyst were slightly greater than were observed with fresh catalyst, while at 752°F the reverse was true. This suggests that there was very little difference between the activities of the fresh and MCH pretreated catalysts and that the differences in activity in the pulse and bench-scale reactors were primarily due to heat transfer effects.

From the experiments conved out thus far it is evident that the resotivity of the MCH-platinum system was considerably greater in the pulse reactor than in the bench-scale reactor. This is shown quantitatively by comparing first order rate constants from data obtained in the two systems. In the pulse reactor the rate constant at 662°F (He) was 53.3 sec⁻¹ (68% MCH conversion; data of Run 196-3, Table 8), compared to 0.60 sec⁻¹ at 842°F in the bench-scale reactor.³) Converting the pulse reactor data to 842°F, using an activation energy of 11.7 k cal/mole,³) gave a rate constant of 176 sec⁻¹ or about 295 times greater than was obtained in the bench-scale reactor. Thus the MCH-platinum system appears to have a potential reactivity about 300 timer greater than was demonstrated in the bench-scale tests.

Presumably the higher reaction rates observed in the pulse reactor were due to heat transfer and diffusion effects that were much less adverse in the pulse reactor system. For example, in the pulse reactor the gas volume of the MCH pulse was about that of the catalyst pore volume (0.2 ml), so diffusion effects would be minimized in this system. Further, the heat of reaction for complete conversion of one microliter of MCH is about 0.55 cal. The heat capacity of the catalyst plus quartz in the pulse reactor is 6.3 cal. Thus the maximum total temperature drop in the catalyst bed per pulse was about 0.55/0.5 = 1.8°C or 3°F. A temperature difference between furnace and catalyst bed of 200°F was observed in the bench-scale reactor at high MCH conversion. This suggests that reactivities possibly 300 times greater could be obtained in continuous reactors if high heat transfer rates and elimination of diffusion effects in catalyst pores could be achieved.

Good catalyst stability was observed at 752°F with hydrogen carrier gas but not with helium. Presumably good stability is obtained because the coke precursors formed during dehydrogenation are reacted from the catalyst surface by hydrogen. This suggests that with helium carrier gas the hydrogen concentration in the catalyst pores due to hydrogen generated by the dehydrogenation reaction, was not great enough to remove the coke precursors, and hence the catalyst was partially poisoned. With hydrogen carrier however, a high hydrogen concentration was present in the pores, and the coke precursors were reacted rapidly from the surface.

Dehydrogenation of Decalin Over Various Catalysts

In earlier work³)18) it was shown that catalysts stable for the dehydrogenation of monocyclic naphthenes, were not necessarily stable for the dehydrogenation of dicyclic naphthenes. Thus UOP-R8 platforming catalyst was quite stable for the dehydrogenation of methylogolohexane³) (MCH), but showed great instability for the dehydrogenation of dicyclohexyl¹) (DCH) and Decalin¹) (DHN). A number of commercial and laboratory-developed catalysts tested in the bench-scale apparatus had good stability for MCH dehydrogenation. It was of interest now, to evaluate a few of these catalysts for the dehydrogenation of Decalin.

The apparatus was a tubular flow reactor equipped with conventional devices for measuring feed flow rates and for collecting liquid and gas products. The reactor was a stainless steel tube (No. 547, 1/2-in. IPS) 32 in. long, 5/8 in. ID, and was heated by an electric furnace. The catalyst was contained in the annular space between the thermovell and the reactor wall. The dimensions were such that the catalyst bed had an annular thickness of 1/16 (one pellet diameter) and a length of 4-1/2 in. (7 ml volume). The complete apparatus was described in detail in a previous report.

Product analyses were carried out by mass spectrometry and by GLC*) from which conversions and selectivities were calculated.

Both zero and first order rate constants were calculated from the rate of disappearance of decalin using the following equations:

sero order:

k, atm
$$\sec^{-1} = \frac{\text{LHSV}}{3600} \times \frac{0 \times 22,412}{\text{NW}} \times \frac{T}{273} \times f$$
 (1)

first order:

k, sec⁻¹ =
$$\frac{1HSV}{3600} \times \frac{\rho \times 22,412}{MW \times P} \times \frac{T}{273} \times 2.3 \log \frac{1}{1-f}$$
 (2)

where

IHSV = liquid hourly space velocity (i.e., volumes of feed/volume of catalyst bed per hour)

MW = molecular weight

P = reactor pressure in atmospheres

T = reaction temperature in *K (reactor wall temperature)

p = liquid density
f = fraction reacted

Relative reactivities and apparent activation energies were computed from the first-order rate constants.

The reactor wall temperature was measured by a thermocouple pressed against the outside reactor wall by the furnace block and located about 1" below the top of the catalyst bed. The catalyst bed temperatures were measured by thermocouples contained in the thermowell. The thermocouples were 1" apart and the top thermocouple was about 1/2" below the top of the catalyst bed. The "effective" catalyst temperature was somewhere between the reactor wall temperature and the catalyst bed temperature. For somputing rate constants and apparent activation energies the reactor wall temperature was used, as this closer to the "effective" catalyst temperature than the thermowell temperature.

Three commerical catalysts and seven laboratory catalysts were evaluated for vapor phase dehydrogenation in the bench-scale reactor at 10 atm pressure and 842-1202°F. These were:

a) 5% Carbovax 1000 on Chromosorb W Column.

P. OFKAN WORDS IN A

Designation
RD-150
PHF-ii
uop-r8
Shell 46 Shell 108 Shell 107A Shell 107B Shell 105B Shell 45 Shell 91A

Seven milliliters of catalyst (10-20 mesh) were used in each test; F-113 DHN was the test feedstock. This Decalin contained 74.64 cis-DHN, 25.0% trans-DHN and 0.4% tetralin (THN).

Some descrivation during the runs was observed with all of the catalysts tested. Thus the catalysts were characterized as to activity by the total Decalin conversion or by the first order rate constant at 842°F, where catalyst descrivation was slight. Relative rescrivity of a particular catalyst then was taken as the ratio of the first order rate constant obtained with that catalyst to the rate constant obtained with the standard catalyst at 842°F (10 stm pressure). The complete data are presented in Tables 60 through 65 in the Appendix.

Results

All of the catalysts were more active than the standard catalyst except RD-150, which was slightly less active (Table 12). Relative activities at 842°F are tabulated in Table 12, and at higher temperatures can be deduced from Figure 8, which is a plot of DHN conversion as a function of block temperature. Overall, the most active catalys. sas the Shell 46, which was also the most stable. Catalyst descrivation with increasing temperature is reflected in the convex shape of some of the curves.

Apparent activation energies were calculated for both zero order and first order rate constants. Because of the extensive deactivation at the higher temperatures with some of the catalysts, only the rate constants at 842 and 952°F were used in this computation. Over this temperature region the apparent activation energies ranged from 6.5 to 11.4 kcal/mole for first order kinetics (Table 12). Lower activation energies were obtained for zero order kinetics because for a given increase in conversion, the first order rate constant increased more than the zero order constant. Figure 9 is an Arrhenius plot of the data in which the lines through the points at 842 and

a) The first order rate constant is given by $k_1 = K/t \log 1/1 - f$ and the zero order constant by $k_0 = (K^*/t)f$ where f = f raction reacted.

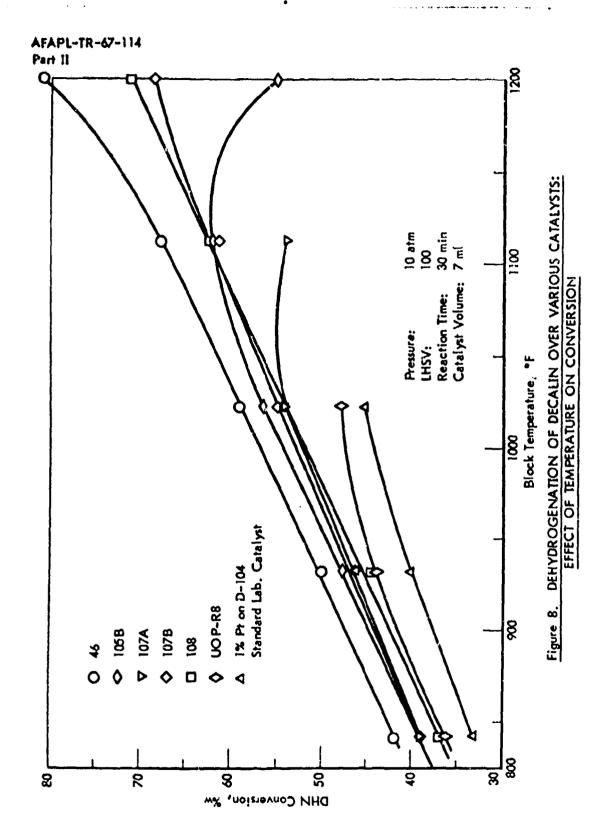
a) At 1112 F

Table 12. DERYDROGENATION OF DECALIN OVER VARIOUS CATALYSTS

and Stabilities	F-113 DHN 7 ml 10 etm 100
Relative Activities	Feed: Catalyst Vol: Pressure: IHSV:

Cetalyst	15 Pt on Karehaw 0104 Al ₂ 0 ₂₅ Standord Catalyst	84-001 100-48	10780-	-08201 -08201	10280- 107A	10280- 1078	10280- 1058	8	8-1-8	出	55.
DHM Conversion, Se, 842 F	32.8	36.2	10.2	37.5	38.9	39.1	39.3	32.5 30.4	30.4	36.2	4.8
First Order Rate Constant, sec.	0°33	0.45	8.3	0.44	0.47	0.48	0.47		0.37 0.36	6.43	6.36
Relative Activity Standard Catalyst m 1.00g, 842°F	86.	51•1	5.		1,21	1.23	1.2		0.95 0.92		±.
Afeax, °F at 842 1022 1202	on 15 .	7 <u>8</u> ·	63 65	11 16 124	16 75 31 48)	= # 8	287	2 %·	7 5	921	5 <u>-</u> .
Eact, kcal/sole Zero Orden, ats sec ⁻¹ First Orden, sec ⁻¹	1.8	7.4 8.7		1	6.1 5.8 7.8 6.5	5.6	7. 88 8.	5	5.6 6.7 11.4	. 2	. 5

- 35 -



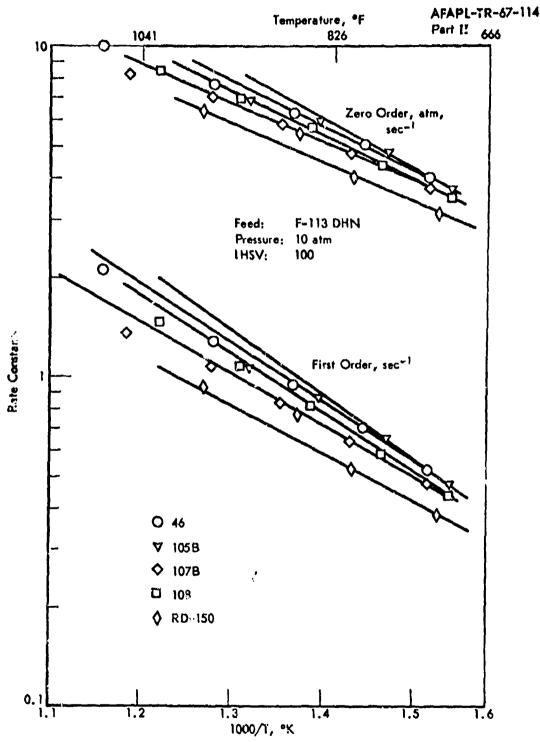
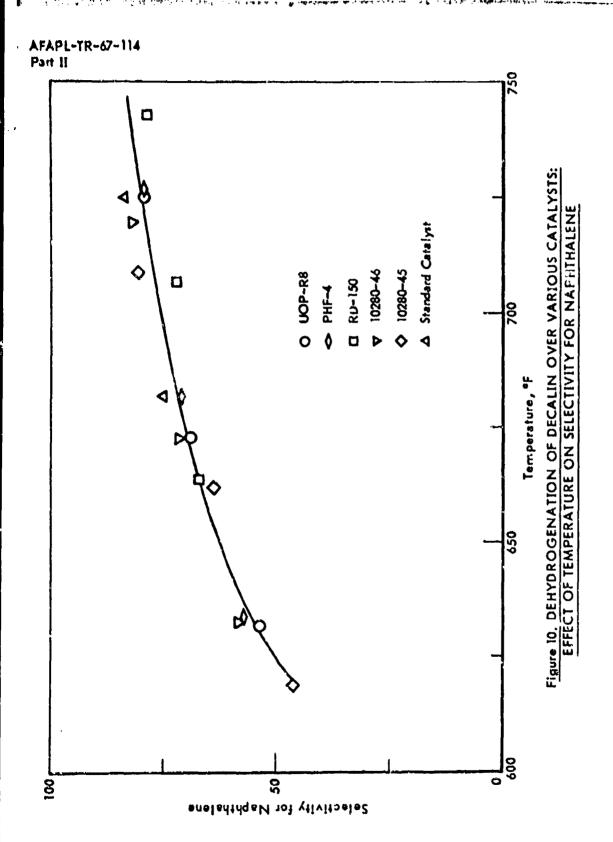


Figure 9. DEHYDROGENATION OF DECAUN OVER VARIOUS CATALYSTS:

<u>TEMPERATURE COEFFICIENT</u>



932°F are extrapolated over the complete temperature region. Deactivation at higher temperatures is evident as the data points fall below the extrapolated curves. The reactor wall temperatures were used for calculating the activation energies.

Selectivit; for naphthalene at a given DHN conversion was different with each catalyst. This appeared to be a temperature rather than a catalytic effect, however, as in a plot of selectivity for naphthanlene as a function of "gas exit" temperature (Figure 10), the points fell reasonably close to a single line. The temperature at the bottom of the catalyst bed was taken as the gas exit temperature.

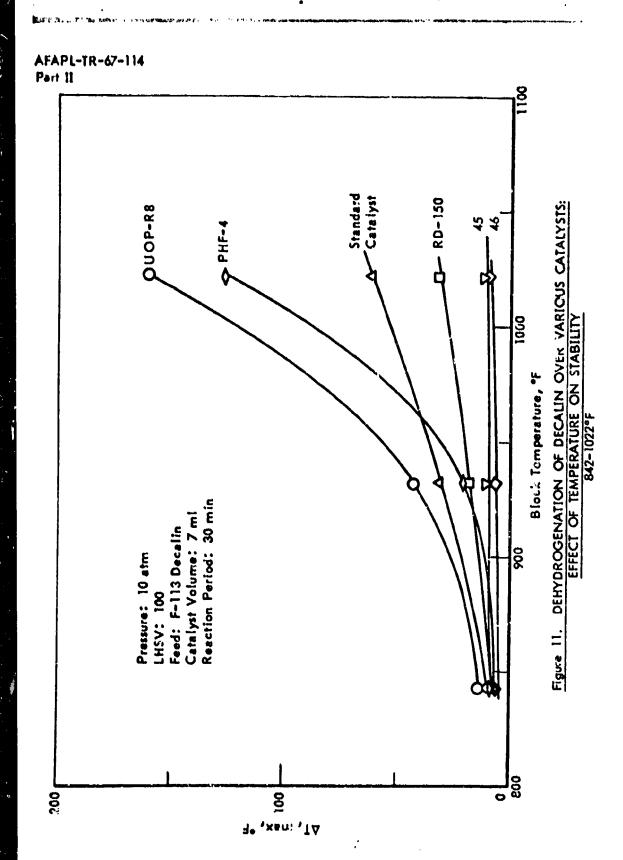
Some deactivation was observed with all of the catalysts tested (Table 12). This is shown graphically in Figures 11 and 12. These curves show the increase in catalyst bed temperature ($\Delta T_{\rm max}$, °F) as a function of furnace block temperature for a series of runs at 842 to 1022°F and at 842 to 1202°F respectively (at 10 atm pressure). The magnitude of the bed temperature increase was taken as a measure of catalyst deactivation. Based on this criteria the catalysts in order of their decreasing stabilities were: 46 > 108 > 1078 > 91A > 1058 > RD-150 > standard catalyst > 107A > UOP-RS. This evaluation depends somewhat upon the test temperature as 1022°F Shell 1058 was more stable than either 1078 or 91A, but was less stable than the latter two at 1202°F. It is worth pointing out that the orly catalyst that had even moderate stability at 1202°F was Shell 46.

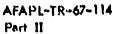
These results are for 10 atm pressure and 30-60% conversion. In previous work it was shown that at higher conversions (80-90%) both the UOP-R8 and the standard laboratory catalysts had good stability at 1022°F. 1) Presumably the other commercial Catalysts would also be more stable at high conversion.

In previous work with Decalin it was observed that catalyst stability was enhanced when the operating pressure was increased from 10 to 50 atm pressure.¹) Thus it was of interest to see if stability could be enhanced at higher temperatures by increasing the pressure to 30 atm.

One commercial catalyst, PD-150 and one laboratory preparation, Shell 107B, were tested over the temperature range of 842-1202°F at 30 atm pressure. With both catalysts, stability was enhanced at the higher pressure. For example with the RD-150 at 1112°F an increase in catalyst bed temperature of only 23°F was observed at 30 atm compared to 117°F at 10 atm pressure. Selectivity for THN + N was about 8-10% lower than was observed at 1022°F (Table 15). At higher temperature the catalyst was moderately stable but there was a considerable decline in both conversion and selectivity for THN + N, and a corresponding increase in yield of cracked products at both 10 and 30 atm. Presumably a hydrocracking-type reaction becomes predominant at 1202°F at both pressures (Table 15).

With Shell 107B at 1112°F only a 9°F rise in catalyst bed temperature at 30 atm (down from 56°F at 10 atm) and at 1202 a 108°F rise (down from 160°F at 10 atm) was observed (Tablell). Selectivity for THN + N at 1202°F was high at 10 atm (92.5%) but only moderate at 30 atm (77.9), and considerably more cracked products were observed at the higher pressure.





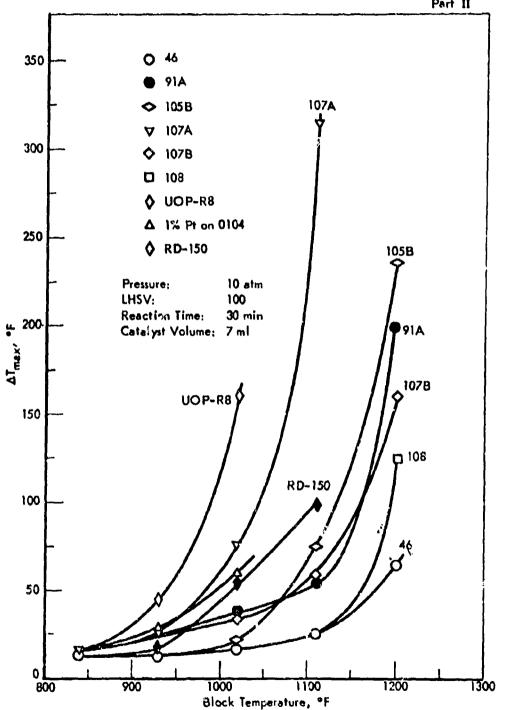


Figure 12. DEHYDROGENATION OF DECALIN OVER VARIOUS CATALYSTS

EFFECT OF TEMPERATURE ON STABILITY: 842-1202°F

Table 13. DEHYDROGENATION OF DECALIN OVER SINCIAIR-BAKER RD-150 CATALYST

Catalyst Volume: 7 ml LKSV: 100 Reaction Time: 30 min

Feed: F-113 Decalin 74.6% cis DE: 25.0% trens DHW 0.4% THN

Reg No.			10346-15					10,08.4					10.17		
Pressure, sta			2					2					12		
Temperature, *P						(L							L		
Elock ·	3	×	2201	या	7502	3	8.	ğ	ZIII	823	2	×	ä	2112	ğ
TITES.	78.×	728-97	346-55	22-926	क्ष्र	8	84-58	15-148	34.96	15-97-1	25.4	5	521-55	r R	13-61
Ontalyst Bed Profile	863.88	28.45	112-ST	1501-116	1112-39 662-75	C62-75	な	73-62	976-372	₹-L11	17-739	スカル コカル	4 1-	7.7	;¥ ::
	9	673-R2	30-16	699-94	1062-1125	44.57.9	6780	87	35.67	367-1135 657-56	65-159	8-4.5	\$\frac{\x}{2}	25.72	9
	04 X 9	200	702-09	82-72 72	1340-1121	# K.9	67-13	91-101	86-92	इट्रा ४५०१	55.25	74.05 78.45	Ä	Tiller 1:32-23	13243
	3	16-299	6:-912	1:7-4:51	1035-112.	4	622.et	72-21	8-EE	1382-1125	6179	27-92	75-5	3	91
ξ	17	ķ	ĸ	lin.	⊽,	2	n	*	æ	.8	4	\$	۰	;;	\$; 6
Product Amilysis, \$4															
THOS DAY	8	¥.	ŝ	0.97	97.2	0.7	₹.0	19.5	25.2	1.22	37.6	8.5	38.1	19.5	1.15
20	9.	*	27.8	27.9	1.9	ş	9,	2	5.15	»; %	29.1	2	19.5	£.9	2).2
ኄ	9.0		1.1	\$.0	11.3	0.0	9.5	1	\$.\$	0.21	6,		0.3	2.9	15.4
Ħ	7.21	10.2	10.3	5.2	2.6	10.9	10.9	5.8	7.5	2.2	23.3	ź	23.2	15.5	2-7
4 .	0.0	0.0	0.0	4.0	3.4	7	7.7	0.1	2.5	3.0	0.7	0.3	0.3	3.5	6.5
*	21.1	Š	8.	1.1	3.7	ੋ -ਕ	8.6	11.2	42.9	3.6	9.5	36.0	8	7.7	4:2
ŝ	7.	0.2	0.5	100	0.1	 6	0.2	9.5	6.3	0.5	0.1	0.1	6.2	2:3	*;
Cracind, 15q.	9.0	0.0	0.0	1.0	9.8	0.0	0.0	0.1	7-7	9.6	0.0	0.0	0.1	77	7. %
Tield TM: %	n.8	9.6	3.9	1.6	2.2	20.5	25.51	8.9	6.9	1.0	2.3	₹.	22.5	;; ;9	2.3
DE Carenton, Y	35.1	\$	51.8	χ, ο.	8	ğ	11.8	52.0	57.0	X0.5	ņ	£.3	53.8	63.0	9.6
Selectivity for TM . H. For	8.5	88	8.	4.7	19.2	8	9 .	8.7	97.5	17.9	; \$	8.1	?:5	8. 8.	39.5
Mite Constants					-										
Zero Grder; atm see 3	3.16	77.	5.49	6.41	8.∻	7,	4.23	8	6.49	5.99	3.10	1.31	5.7	2.3	8
Mrst Order; see 7	٠,٣	0.53	0.77	80	\$	P. 9	0.55	67.0	9.9	L4.0	0.13	0.13	0.27	6.5	9
Lace, beal/onle	6.8		•	•	-	←−6.7 →	ĵ	•	•	•	- 0:	֝֟֝֝֟֝֟֝֝֟֝֝֟֝		- ,	,

a) Marian increase in catalyst bed temperature during the 30 staute run.
b) inclemitified.
c) for significant as cold spot was moving done the catalyst band.

Table 14. DESTONGEMENTION OF DECALIN OVER SHELL LOTB LABORATORY CATALYST

Catalyst Volume: 7 ml. LMSV: 100 Resotion Time: 30 min

Feed: F-113 Decalin 74.6% cis Din 25.0% trans Din 3.4% Tin

Run Ko.			26-27601	26				10548-21		
Pressure, ate			2					R		
Toperature, F										
Block	842	932	1022	2111	1302	842	932	1022	1112	120
1187	725-30	797-99	14 PS	646-50	1054-87	7.0.7	25.00	35.1	9	מר ליוטו
Catalyst Bed Profile	624-35	28-299	714-48	77.433	354-1114	779	2	700-12	20,00	01/1-1/10
	71-909	15-669	582.94	2	842-957	979	129	869	72-21	847.87
	503-15	15-759	26-689	736-43	831-67	653	33	202	7.65	831-44
7	619-22	1651-60	3078	756-54	842-44	999	693	2	765-51	855.54
47 asx, **	=	<u>-</u>	7	58	5	-	-	•		2
Product inalysis, Se					!	•	•	, 	•	3
trans CEX	۳. ۳.	26.4	21.6	12.0	12.2	53.7	39.7	80	2,2	12.5
cis DHR	23.6	26.8	24.4	21.4	21.3	12.7	12.3	7	20.2	12.5
(₀)	3	0.0	90	20	80	0.0	0.0	0,0	0.2	9.7
	18.5	3	9.7	2.0	2.5	24.6	33	23.5	21.7	
(4)	9.0	0.0	0.0	0,0	3	-0	-0	0.3	0	o
× '	9.0	32,3	8: **	5,3	56.3	80	17.6	900	15.1	53.5
	0.7	0.2	0.2	2.0	3.0	0	0.2	9.2	0.5	6
Creeked, 114.	00	00	00	0.3	3.7	0.0	0	0	-	=
Tield THM, Je	189	13.9	80	25	***	21.3	8	2	~	4
DHN Cornersion, Va	8	16.7	24.2	2	8	2	8 27		; &	2
Selectivity for DE . N. Je		29.6	8	8	3 8	7 8	9	3 8	3 4	2
•					}	;	3	2	? pr	<u>:</u>
lero Order; ata see	3.17	2	5.82	7.91	23	2	7	7	63 (6
First Order: sec 7	0.48	19. 10.	78.0	2	35	2 5	3 5	5 6	3 5	5 6
F. A. Seal facto	-		•			?	77.0	75.0	3	Š
	,				9	Ĺ				

a) Mariana increase in catalyst bed temperature during the 30 sinute runab) Unidentified.

Thus it appears that at 1202°F increasing the reactor pressure will enhance the stability of the catalyst, but that selectivity for THN + N and in some cases DHN conversion will be reduced appreciably. Further, it appears that at these elevated pressures and temperatures a hydrocracking-type reaction becomes none important resulting in increased yields of cracked products.

The two most stable catalysts tested thus far for the delydrogenation of decalin were the laboratory catalyst. Shell 46 and Shell 108. Both catalysts had comparable stabilities at 1112°F but at 1202°F the Shell 46 has more stable. With these catalysts selectivity for THN - 1 has high at 1112°F and lower temperatures (90+5), but decreased by about 5-105 at 1202°F. For a given weight of platinum stability varied inversely as the pore size of the support. Increasing the operating pressure from 10 to 50 atm increased catalyst stability but at 1202°F decreased selectivity for THN+N and in one case decreased the Decalin conversion.

The Shell %6 was also the most active catalyst tested thus far and was 1.28 times more active than the standard laboratory catalyst.

Effect of Pore Volume on Catalyst Stability

In a previous report it was suggested that catalyst instability was due at least in part to the pore structure of the catalyst and that greater stability would be obtained with catalysts containing smaller pores. 19) The everage pore dismeters of five catalysts were calculated from the surface and pore volumes and are tabulated in Table 15. These catalysts continued 0.7% to 1.0% platinum on various alumina supports; their physical properties are tabulated in Table 16. Figure 13 is a plot of catalys' bed temperature increase (ATmax, F) as a function of average pore diameter and shows indeed that better catalyst stability was obtained with catalysts having smaller pores. (The Shell 45 was not included as the chemical composition of this catalyst was quite different from that of the other five catalysts.) While the least stable catalyst had the largest pore dismeter and the most stable catalysts had the smallest pore diameter, there is an anomoly in the region of 100 A pore diameter. Thus the PHF-4 and the standard laboratory catalyst have about the same pure diameter but the catalyst bed temperature increase with the former was about twice that observed with the latter catalyst. As pore distribution for these two catelysts are about the same (Table 16), it ampears that pore size is not the only factor controlling catalyst stability. This is being investigated further.

As mentioned in the previous reports it is believed that catalyst stability during the run is due to hydrogen, generated by reaction, reacting with coke precursors on the catalyst surface. Presumably in smaller pores the hydrogen partial pressure will be greater due to a higher surface to volume ratio, and hence a greater fraction of naphthene converted per unit time. This higher hydrogen pressure causes more rapid reaction with coke precursors and hence the catalyst appears to be more stable.

Dehydrogenation of Decalin: Diluted-Bed Reactor

A study of the kinetics of the dehydrogenation of Decalin has been initiated to obtain data for use in developing a computer program for this

Table 15. DEPURCEMATION OF DECALIN OVER VARIOUS CATALYSTS

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81
81
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431

Feed: F-(13 DAM
Catalyst Volume: 7 mf
Freshure: 10 sta
UNSY: 100

	278	8427 Block Teapersture	aparature	Total	Myrs T.	1
Catalyst	Cone .,	Rate Constant, sec-1	Latetive Lasetivity ³ !	Toing.	et 1022 F Block Yesp	Average Pare Diameter, A
15 Pt on Harsham 0104 AlgOs ⁸)	37.8	0.33	8,	0.286	=	8
Sinciain-Baker 20-150; 0-655 Pt	30.4	0.36	0.92	0-405	Ä	ä
IS Pt on Alada	10,2	05°0	82.1	0.25!	624	43
Spell 45	8,14	35. 0	3.	מבס	=	ತ್ತ
American Cyannaide American !!E-4; 0.8; Pt	36.2	0.43	9.1	0.607	ĸ	## ##
10° 48; 0.76 Pt	36.2	0.45	1.15	0.719	8	175

s) Standard Imboratory catalyst.
b) Standard Imboratory catalyst = 1,00.

Table 16. PHISICAL PROPERTIES OF WRIDES CALAINSTS

Catalyst		13 Ft 98 0:04 A: ₂ 0, (Standard	American Cramatida American FF-4; 0.85 Pt	Sincleir-Baine 10-15; 0.55; Pt		15 Pt 8	10280-45		(\$ Pt on
		Catalynt							2 502.7
Surface Area, 12/9		8	222	63)		23	115		791
Bulk Density, g/cc (10-20 mesh size)		96°0	8. 0	0.93		8.0	9.88		0.63
Total Pore Yolume, cc/g		0.788	0.837	0.402		0,23	0.377		0.7:9
Pore Size Distribution	Pere Disseter, A		Cumistive Volume, cc/g	ec/8	Pare Dissetor,	Concletive Yoluae, cc/g	Folume,	Pore Disseter,	tive fot
	•	0000	0000	0,042	63	210.0		24	0.033
	~	9900	800	190°0	22	0.027	9300	35	\$20°0
	5	88.0	0.00	5500	S	6.17	0,013	23	050.0
	*	, 3	9100	0.14	25	92.0	0.075	23	6.0
	83	0.013	2300	5.0	185	0.219	0,312	147	981-0
	2	0.085	0.193	0.255	354	0.73	0.33,	235	0,378
	ž	0.192	0.526	0.315	2	•	0.330	5:3	0.533
	757	•	•	0.311	22	0.25!	0,327	2	0.850
	8	0.266	0.83	0.402					
a) Pore distribution de	starained or	determined on R-S alumina hase	hase						-

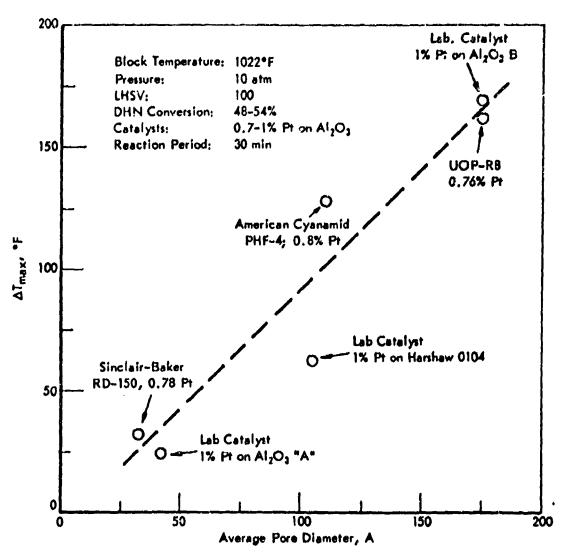


Figure 13. DEHYDROGENATION OF DECALIN OVER VARIOUS CATALYSTS:

EFFECT OF PORE SIZE ON CATALYST STABILITY

resotion. For this work the catalyst was highly diluted with inert material. the reactor was operated at low conversions, and the apparatus was modified so as to try and maintain isothermal conditions within the cutalyst bed as much as possible. This modification consisted of removing the 1/2-in. OD thermovell from our 5/8-in. ID reactor tube and substituting two 1/8-in. OD thermovells, one at the top and one at the bottom of the reactor tube. The top thermovell extended down 1-1/2 in. into the catalyst bed; the lower thermovell just touched the bottom of the catelynt bed. Figure 14 is a drawing of the modified reactor tube.

Results were obtained at 707 to 797°F; 10-50 atm pressure: IRSV's of 350-1400 with our standard 1% Pt on Al₂O₃ catalyst. 0.5 ml (0.48 g) catalyst was used and the catalyst was diluted with Cu pranules and/or quarts chips to give a total bad volume of about 29.5 ml and a bad length of about 5-1/2 inches.

The procedure for carrying out the runs enalyzing the products and calculating the rate constants were described in the previous section of this report.

The kinetics of the dehydrogenation of Decalin involve rates of: a) isomerization of cis and trans Decalin; b) dehydrogenation of cis and trans Decalin and of tetralin; c) dehydrogenation of tetralin. Thus for our study six different feeds were used that contained various amounts of cis and trans Decalin and tetralin. Hydrogen was added to those feeds containing tetralin in about stoichicmetric amounts to simulate product material from Decalin dehydrogenation.

The feed compositions were:

Table 17. DEHIDROGENATION OF DECALIN AND TETRALIN

Feed Compositions

Feed Number	1_	20)	<u></u>	45)	50)	6_
Liquid Composition, for						
trans-DiN 01s-DHN	91.0 8.8	57.0 42.5	25.0	43.6	11.8	0.1
THI	0.0	0.2			51.8	
N	0.0	0.0		0.6	0.4	0.8
Others	0.2	0.4	0.0	0.6	0.5	0.6
H_2 Added, moles $H_2/mole$ THN	0.0	0.0	0.0	5.6	2.6	3.0

Feed 2 was 50% Feed 1 and 50% Feed 3.

Feeds 4 and 5 represent an approximate product mixture after dehydrogenation Decalin to 50% conversion: Feed 6 represents a product mixture of 100% conversion of Decalin to tetralin.

b) Feed 4 is 50% THN end 50% Feed 1. c) Feed 5 is 50% THN end 50% Feed 3.

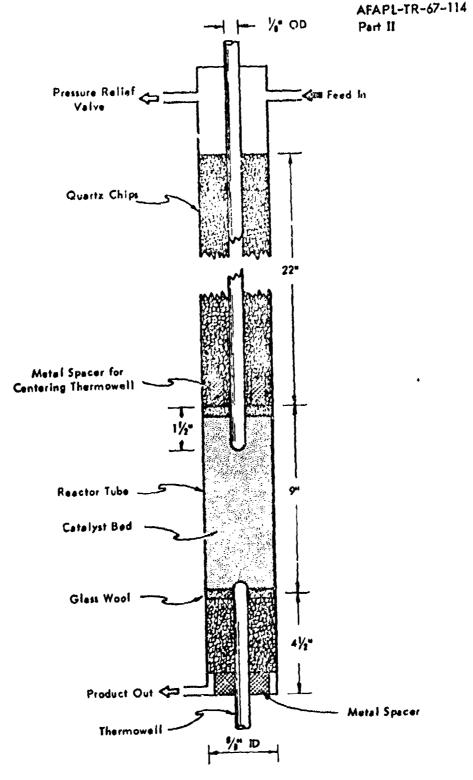


Figure 14. DILUTED BED REACTOR

AN MATRAGY-114
Part II

The effect of process variables on reaction rates and product distribution are discussed in this section. The utilization of the experimental results for developing a computer program are discussed in another section (see page).

Results

Under our test conditions the dehydrogenation of DHN to naphthelene (N) was a two-step process with tetralin (tetrahydronaphthelene; THN) as an intermediate product. Schematically the reaction can be represented:

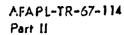
$$\begin{array}{c|c}
\hline
 & I \\
\hline
 & I \\
\hline
 & I \\
\hline
 & II \\
\hline
 & Naphthalene
\end{array}$$

Both resctions were equilibrium limited at high conversions and in the lower temperature region at both 10 and 30 ctm pressure. Reaction II was faster than I. Figure 15 shows the thermodynamic equilibrium constants for Reactions I (Kp_1) and II (Kp_2) as functions of temperature. Kp_2 was obtained by extrapolating the equilibrium data of Allam and Vlugter. 21 Kp_1 was obtained using the data of Miyazawa and Pitzer 22 and the calculated Kp_2 . Figure 16 shows equilibrium concentrations of DHN, THN, and N at 10 and 30 atm pressure as a function of temperature using the calculated Kp_1 and Kp_2 .

A preliminary series of experiments was done with F-115 Decalin (Feed 3; 74.6% cis DHN) at 707 to 797°F; 10-50 atm pressure. In this series the runs on different charges of catalyst did not give reproducible results. For example at 752°F (block temperature) LiSV of 1400 and 10 atm pressure, DHN conversion of 6.8 and 10.5% were observed for two different runs (Table 13, Runs 61 and 50-2). Presumably this nonreproducibility arose from catalyst surface contamination during catalyst pretreatment. However within an one run the catalyst was fairly stable. Thus under the same reaction conditions at 792°F only a few percent change in conversion was observed over a one hour period (Table 18, Runs 74-2 to 75-1). Increasing the space velocity at constant block temperatures gave a slight increase in reaction rate, which suggests that even at LHSV of 700 there was still a heat transfer effect within the catalyst bed. The effect of pressure was similar to that observed previously namely that with increased pressure (10 to 50 atm) conversion was not affected significantly but the rate constant declined markedly with increased pressure (Table 19).

In rune of different charges of catalyst (i.e., A, B, or C, Table 18) the absolute values of the rate constants at a given temperature were different, which suggests that the catalyst was poisonel prior to the start of the runs. With only 0.5 g catalysts small amounts of contaminents can poison an appreciable amount of catalyst surface. Thus the following changes were made in the experimental procedure to reduce the possibility of catalyst contamination.

1. The fresh cutalyst was reduced in situ with hydrogen with heating at 1022°F for one hour.



L Land

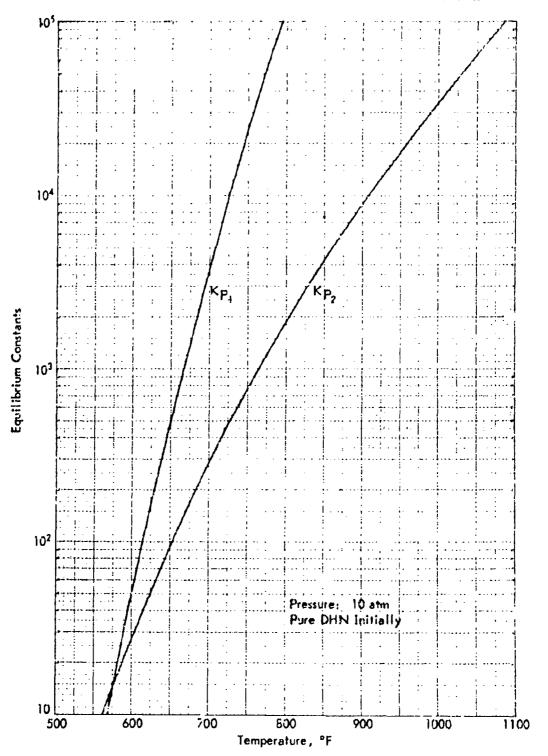


Figure 15. EQUILIBRIUM CONSTANTS FOR DECALIN SYSTEM

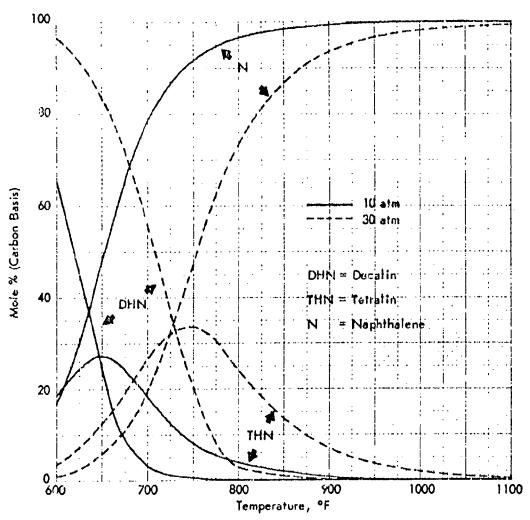


Figure 16. EQUILIBRIUM COMPOSITION OF DECALIN SYSTEM

(..

Table 18. DEHYDROGENATION OF DECALINE DILLTED BED REACTOR

Conversion	
Velocity on	
and Space	
Temperature	
Effect of	

u.
15 ft on Al ₂ 0 ₃ 0.5 st 10 sts
Catalyst: Catalyst Yolune: Pressure:

F-113 Decallin	cis-CHN	trans-CHX	TH
F-113	74.62	25.0,	0.6
Fast.			

Run Ho. 10342-	70-1	70-5	71-1	12-22	73-1	≍ -:	14-2	74-3	14-4	75-1	53-1	59-2	8	156	1-29	2-29	23	3	8
LKSY	200	0053	233	<u>\$</u>	8	8	<u>\$</u>	8	8	8	8	8	ğ	- 8	S	8	8	8	8
Catalyst Change				1	74	1	.	.	1	1		-		- 1 (ag	-		1	P.	1
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Catalyst Bed										:		3	:	5	2	7:	=	2	=
100	•	•	•	•	1	•	•	•	•	,	6.09	103	1072	740	2	787	702	702	2
1-1/2 in. Seice Top	673	88	202	714	741	752	757	755	76!	161	•	,	? ,	? (<u> </u>	3 1	Ś	
Bottos	8	675	23	712	753	252	755	758	255	35	678	673	712	716	10.	75.	747	655	8
Product Analysis, 72												<u></u> .							
trans-DE	26.7	25.5	25.8	24.9	24,9	24.3	26.4	27.6	21.7	77.16	2 2	25 F	25.0	, X	ני	21.7	7 76	15 36	3,
CIS-DER	62.6	0.83	1.19	67.6	8	66.2	67.0	67 3	7.79	88	7	2 69	3	3 0	, ,			3 5	
ZEL .	3.6	5.3	3,6	2.9	7	3-	2.8	a,	,	α,	, ,	3 6	, ,	,	,		4	7 .	7.
200		3.5	7.6	9	8	9	00		7		•	2 6	9 0		,	• •	9 4	;	
Heavier than N	0.0	0.0	3	0.0	-	0	0	0	5	. 7	- 6	, 0	3 -	. 6	7		9 9		
Yield THR, Je	3.1	1.5	2,55	2.5	3.9	1.2	2.4	2.4	2.2		2.5	4	3.6	2,1	5.2		2.2	3.0	7.7
ORK Conversion, Ve	7.01	5.5	13,0	7,5	16.1	9.5	3.5	80	80 72	2	9.7	8	12.2	60	75.8	13.5	7. B	-	10.5
Tirst Order Rate Constant, sec_1	ព្រះ	9.74	0.94	50.	1.21	క్త.	1.3	5.58	1.24		0.67			76.0		20.0	27	8	7

a) Catalyst ollusted with 25 ml Cu and 6 ml quartz chipm. b) Catelyst diluted with 23 ml of Cu granules.

Table 17. DEHYDROGENATION OF DECALIN: DILUTED BED REACTOR

Effect of Pressure

Feed: F-113 Decclin 74.6% cis-DHN 25.0% trons-DHN 0.4% THN 1% Pt on Al203 Catalyst: Catalyst Volume: 0.5 ml LHSV: 1400 Block Temp: 792°F

Reaction Time: 15 min

Catalyst diluted with 23 ml Cu and 6 ml quartz chips

Run No. 10342-	76	77	78-1	78-2
Pressure, atm	10	50	30	10
Temperature, *F Wall Catalyst Bed	779	רודו	765	779
1/2 in. Below Top 1 in. Below Top 1-1/2 in. Below Top Bottom	777 772 · 763 758	770 761 758 758	756 736 732 752	779 768 761 756
Product Analysis, w trans-DHN cis-DHN THN N	24.4 68.1 2.7 4.8	25.3 66.8 3.0 4.9	26.8 64.7 4.5 4.0	24.5 67.4 2.8 5.3
Yield THN, ww	2.3	2,6	4.0	2.4
DHN Conversion, Sw	7.5	7.9	8.4	8.1
First Order Rate Constant, sec-1	1.08	0.55	0.40	1.17
•		i	1	l

- 2. The Cu granules were carefully cleaned by repeated washing in hot isopropyl alcohol until they were dust-free.
- 3. The length of the heated lead was increased from 3 to 5 feet, to insure complete vaporization of the Decalin prior to entering the reactor.

Reproducible results were obtained after adopting the above changes.

An extended series of runs was made with F-113 Decalin (Feed 3; 74.6% cis DHN) at 707-797°F, LHSV of 700 and 1400 and at 10-20 atm pressure. At the lower pressure DHN conversions ranged from 11% to 21% at LHSV of 700 and from 6% to 11.5% at 1400°F (Table 20). The catalyst bed was not isothermal and temperature differences at 16°F at 6% conversion and 54°F at 21% conversion vere observed between the top and a point 1-1/2 in. below the top of the bed. The data were reasonably reproducible and at any one temperature and space velocity the maximum variation in rate constants (3 runs) was 112% and the closest agreement was 16%. At a given temperature the average rate constant (3 runs) was slightly higher at the higher space velocity. This suggests that even at these high space velocities the reaction rate is limited by the heat transfer rates. Activation energies ranged from 9.2 to 12.2. kcal/mole and were slightly higher for first order kinetics (Table 21). Figure 17 is an Arrhenius plot of the data. As was observed in previous work, there was some cis to trans-isomerization during the runs at the lower temperatures as shown by the increase in trans-DHN concentration in the product compared to that in the feed (Table 20).

With increased pressure (10-25 atm) at 752°F, conversions were reasonably constant at any one space velocity (Table 22, Figure 18). However the first order rate constants declined markedly with increased pressure, while the zero order constants increased slightly. This suggests that the reaction order with respect to Decalin is closer to zero order at these low conversions. Figure 18 shows the average values of three runs for conversions, zero and first order rate constants as functions of pressure. Again cisto trans-isomerization was observed that increased with increasing pressure (Table 22).

The approximate reaction order may be calculated from the data of Table 22 by the following method. Assuming that the rate of dehydrogenation is proportional to some power of the pressure (i.e., concentration) then

$$r = kp^n$$

At low conversion the rate is equal to the fraction reacted \mathbf{f} , per unit time, \mathbf{t} , and

$$r = \frac{f}{t} = kp^n$$

In a flow system t is the residence time. For two different pressures at the same space velocity (i.e., constant residence time)

$$\mathbf{r}_1 = \mathbf{f}_1 = \mathbf{k} \mathbf{P}_1^n \qquad \qquad \mathbf{r}_2 = \mathbf{f}_2 = \mathbf{k} \mathbf{P}_2^n$$

e) See footnote on page 25.

Table 20. Defydrogenation of decalin; diluted bed reactor

Effect of Temperature

Catalyst:

Catalyst Volume:

Catalyst Volume:

Catalyst Weight:

Catalyst Weight:

Catalyst Size:

10-20 mesh

Pressure:

10 atm

Catalyst diluted with 23 ml Cu granules and 6 ml quartz chips

F-113 Decalin 25.0% trans-DEN 74.6% cis-DEN 0.4% TEN

Catalyst Charges)	4	ø	U	⋖	ø,	υ	◄	•	υ	4	an	υ	4	#	υ	₩.	æ	υ
Run Ko. 10342-	130-1	135-1	142-1	131-1	136-1	142-2	1.2.1	1-721	163-1	132-1	157-1	144-1	133-1	1.26.1	7.44	17	139-1	145-1
ASRI		- 282	1	Į	1,400	Î		02.	1		8	1		28	1		Q H	Î
Temperature, *P		- 707 -	1		702	1		<u>ا</u> م	1		82	1	1	157	†		757	1
Block Wall Catalyst Bed Profile	929	88	8	9 2.	8	88	ź	52	ğ	22	8	Ę	\$ <u>7</u>	32	192	192	į.	58
Metance From Top, in. 0 1/2 1 1-1/2 Ection	\$\$£8£	38388	5482E	*5888	88885E	83883	\$\$\$\$\$	£434£	2322 5	\$8 5 55	55555	वस्यवद्ध	82222	\$E558	86888	28842	£84.28	\$44.85
Product Arelywis, for trans-Did cis-IEM TIQ Ub) H Up)	2 8 4 0 0 0 5 5 0 0 0	6.4.4.0	7.3.4.0.n.c.	25.8 66.0 5.0 5.0 0.0	8.83	67.9	%%	%7.4.0.1.0 5.00001	%8%.04.0 40.0001	6.000	48 00 00 50 00 00	55.9 5.9 5.0 5.0 0.0	24.7 5.5 1.7.1 1.7.1	1,8% 0 % 0 1,8% 0 % 0	87.50 0 17.0 81.46 0 4	\$ 55 N 0 0 0	25.0 6.0 6.0 1.0 0.0	110 40 40 2 2 2 4 6
Tield THE, &	*;	3.2	3.6	2.7	1.1	17	1.1	3.6	1.4	5.6	2.1	2.3	5.1	5.8	9	3.5	2.5	0.0
IBN Conversion, 🐿	13.7	ii	0.51	7-7	5.9	6.2	16.9	15.4	15.3	4.6	3.0	8.1	9.6	0.81	18.9	11.5	9.6	3.
Selectivity for M, 🐿	67.8	7.0	69.2	65.4	69.5	6:52	77.8	3.3	71.9	2.5	73.8	4 .6	73.0	78.8	23.6	69.0	0.4°	68.3
Rate Constants Zero Order, stm sec ⁻¹ First Order, sec ⁻¹	8.6	3.784	0.827	1.089	9.7.6	7.9 0 883	1.238	1.120	101.1	12.0 1.276	1.099	1.123	1.59	1.361	13.0	35.7	133	1.504

a) The runs in each Series (A, B, or C) were OrL: In different changes of catalyst. The runs in any one series was done on the same charge catalyst.
b) Unidentified.

Table 21. DEHYDROGENATION OF DECALIN: DILUTED-BED NEASTOR

Apparent Activation Energies

Catalyst:

1% Pt on Al203

Catalyst Volume: 0.5 nl Pressure: 10 atm

Temp Range: 707-791°F Catalyst dilutel with 23 ml Cu granules and 6 ml quartz chips

Run No. 10342-	1	30	1	35	1	42
THEA	700	1400	700	1400	700	1400
Eact, kcal/mole						
Zero Order	9.7	9.2	10.1	10.8	10.7	11.8
First Order	10.7	9.7	10.6	11.6	11.3	12.2
First Order	10.7	9•7	10.6	11.6	11.3	ŀ

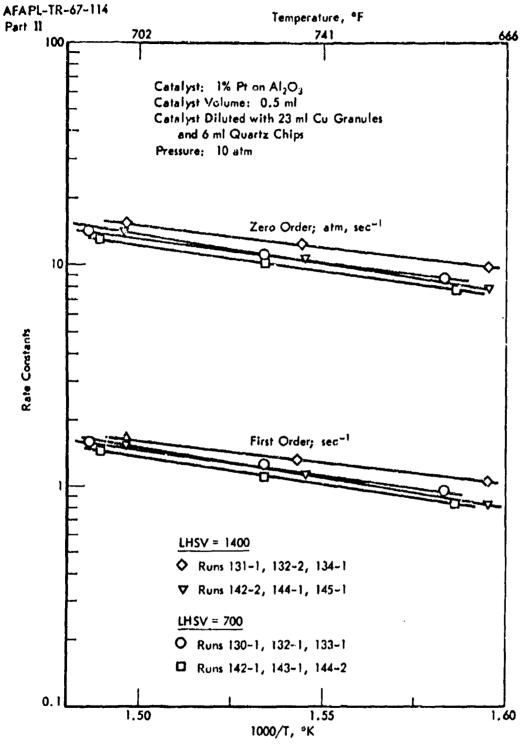


Figure 17. DEHYDROGENATION OF DECALIN: DILUTED BED REACTOR
TEMPERATURE COEFFICIENT

63808

Table 22. DEHIDROGENATION OF DECALIN: DILUTED-HED REACTOR

Effect of Pressure at 752°F

Block Temperature: 752°F Catalyst: 1% Pt on Al₂O₃ Block Temperature: 75: Catalyst Volume: 0.5 ml
Catalyst Weight: 0.48 g
Catalyst Size: 10-20 mesh
Catalyst diluted with 25 ml Cu granules and 6 ml quartz chips

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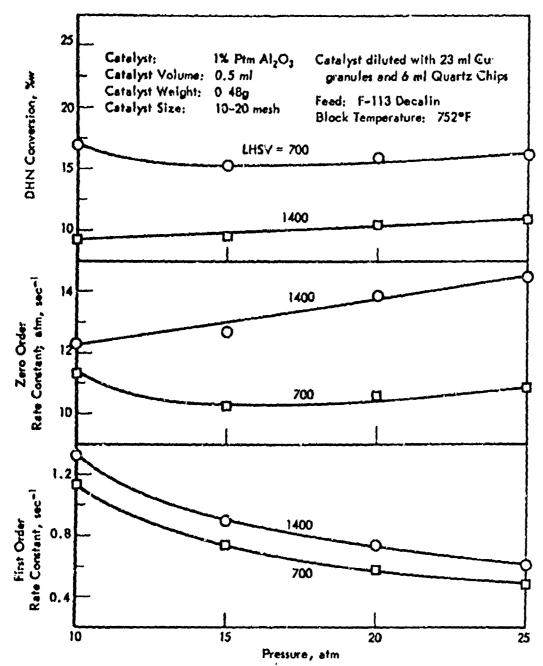


Figure 18. DEHYDROGENATION OF DECALIN: DILUTED BED REACTOR EFFECT OF PRESSURE

n then is equal to

$$n = \frac{\log \frac{\Gamma_1}{\Gamma_2}}{\log \frac{P_1}{P_2}}$$

Using the average conversion values in Table 22 10 and 20 atm, IHSV = 1400

$$n = \frac{\log \frac{10.5}{9.5}}{\log \frac{20}{10}} = \frac{0.043}{0.301} = 0.14$$

Thus the kinetic order of the dehydrogenation of Decalin reaction is about 0.14 the power of Decalin or close to zero order. This is only an approximation however as the concentration of tetralin and hydrogen may also affect the rate.

In this series of tests the reaction did not appear to be equilibrium limited as the product concentration of tetralin and naphthalene were less than the equilibrium values (Figure 16).

In another series of runs, less extensive tests were carried out with all six feedstocks. The data for those feeds containing only Decalin are presented in Table 25, and for those feeds containing tetralin are presented in Table 24.

With the feeds containing only Decalin (Nos. 1, 2, 3), reactivity appeared to be a function of isomer concentration. Thus the feed with the highest cis isomer concentration (No. 3) was the most reactive and the feed with the highest trans isomer concentration (No. 1) was the least reactive (of conversions or the rate constants). This isomer effect on Decalin reactivity was observed at higher overall conversions in previous work. With any one feed the reactivity of the cis isomer was greater than that of the trans when the cis concentration was over 50% (Feeds 2 and 3), but with a high trans DHN concentration (Feed 1) the reactivity of the cis isomer was greater at low space velocity (700) but at high space velocity the reverse was true. Why space velocity should effect the relative reactivities of the cis and trans DHN isomers is not clear, but it does not appear to be a temperature or conversion effect.

Overall activation energies for the first dehydrogenation step were inversely proportional to reactivity and were 12, 20, and 22 kcal/mole for Feeds 3, 2, and 1 respectively. With all three feeds pressure did not appear to effect conversion appreciably.

Some isomerization occurred concurrently with dehydrogenation. For example with Feed 3 (74-6% cis) in four of the runs there was more trans DHN in the product than in the feed, indicating cis to trans isomerization. With the high trans feed however (No. 1; 91.0% trans) the amount of cis in the product was greater than that in the feed in only one run. This suggests that the rate of cis to trans isomerization was greater than that of trans

Table 23. DEHYDROGENATION OF DECALIN MIXTURES

Catalyst Diluted With Copper Granules, mi Quarts Chips, ml Total Bed Leng, , inches
15 Pt on A1203 10-20 0.5
Catalyst Sise, mesh Catalyst Volume, ml

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Pet Bake							-							_			~						~			_
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to cis, although the rate of the latter reaction may have been equilibrium limited as the percent cis at equilibrium at 700°F is about 114 (Figure 19).

Pure tetralin was 3 to 4 times more reactive than Decalin (Table 24). Quantitatively base on first order rate constants, THN was 2.8-3.1 times more reactive than cis DHN (Feed 3) and 3.3-3.9 times more reactive than trans DHN (Feed 1). Tetralin conversions ranged from 15 to 32% with high selectivity for naphthalene (90-99%). Conversion of tetralin to Decalin was less than 1% except for the run at lowest temperature IHSV and highest pressure (1.e., longest contact time) where about 10% of the tetralin reacted was converted to Decalin (Run 164-1). The activation energy (first order constants) was at 15 kcal/mole, which was somewhat lower than the 24 kcal/mole calculated earlier from Decalin dehydrogenation data. As was observed with Decalin, pressure did not affect conversion.

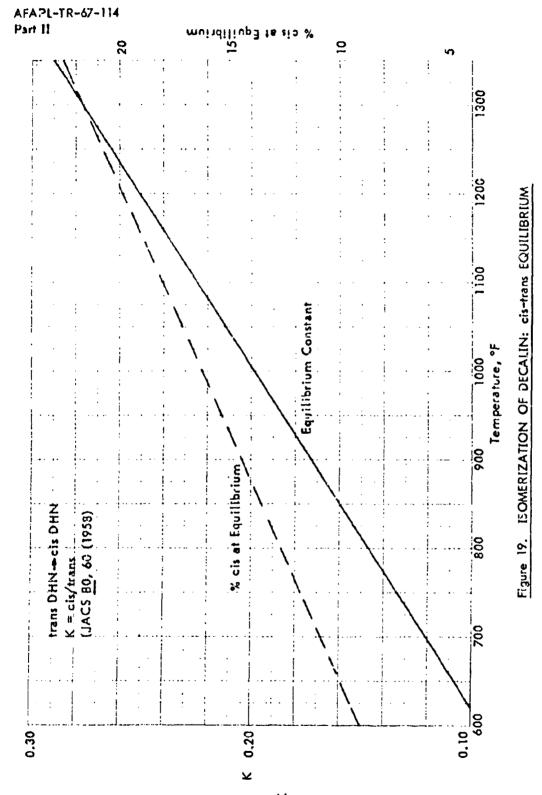
When mixed with Decalin the relative reactivity of tetralin was greater than was observed with the pure components. Thus when mixed with cis Decalin (Feed 4) based on first order rate constants the reactivity of THN was 4.5-5.5 times that of Decalin (Table 24) while with a trans DHN mixture (Feed 5) the reactivity of THN was 7.7 times that of Decalin. Activation energies for dehydrogenation of THN and DHN were lower in the mixtures than were observed for pure components.

Summery

The results obtained with Decalin and tetralin over a diluted catalyst bed at high space velocities generally were in agreement with those obtained in earlier work with ar undiluted catalyst bed (7 ml catalyst; IHSV of 100); 1) namely that cis-Decalin was more reactive than trans for both dehydrogenation and isomerization. However for dehydrogenation in the diluted catalyst bed the value of the first order rate constant at 752°F, 10 atm, (1.16 sec-1) was about twice that obtained under standard testing (0.63 sec-1) and the activation energies (10 atm) were about 28% to 60% higher with the diluted bed technique. This suggests that the rate of reat transfer to the catalyst was greater in the diluted bed-high space velocity configuration which in turn lead to higher reaction rate. These results support a previous conclusion namely that during the earlier bench-scale tests the rate of heat transfer from the reactor wall to the catalyst bed was a limiting factor in the overall rate.

Pure tetralin was more reactive than Decalin for dehydrogenation and the rate of dehydrogenation of tetralin was greater than the rate of hydrogenation. The dehydrogenation of both Decalin and tetralin were about zero order in hydrocarbon. At low conversions activation energies appeared somewhat higher than those obtained in earlier work, possibly due to better heat transfer at higher conversions and temperatures. In Decalin-tetralin mixtures the relative reactivity of tetralin was greater than was observed with pure components.

Additional work will be done on the Decalin-tetralin system only if needed for the development of the computer program for this reaction.



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Table 24. DEHYDROGENATION OF DECALIN-TETRALIN MIXTURES

Catalyst: 1% Pt on Ai₂O₃ Carelyst Size: 10-20 mesh 2: Catalyst Volume: 0.5 ml 6

O3 Catalyst Diluted With:
esh 23 ml Copper Granules and
1 6 ml Quart. Chips
Total Bed Length: 5-1/2 in.

feed No.									ľ			1			١			ſ
Run No. 20548.	151-1	151.2	152	17.51	153.2	3	158.1	5.82	553	160-1	160-2	135	1.43	2.491	, Sã	14	3	15
fred, &. (Rfree) trans_DEM			9.5			1		1	T :			1	†		T ;		1	1
ST.			Š			1 }			, z			1 1			2.4			11
Gherr	11			× 8		+			ر ا ا ا			1			200			H
Ho/THM mole ratio			9				ļ		9			1 1			5 I	+ 		11
Pressure, etc.	ឌម៌	ဒ္ဌ	2 8	28	88	RB	83	22	28	28	83	8 }	8.8	2 8	S 2	0.4	8 3	1
Temperature, ?	1	. 44.	1	- (י ק	-	- {	. !		-	- A	. ,	-	- 7		•	- . î	
Mall Joseph Pres Ton of Bal	Ę	\$	Š	Z.	3 g 	兔	\$	ξ ξ . –	3	55	3 E	E	ğ	2€	, ŝ	77	្រ ទួក់	 ÷
75 mm 2/	116	116	71,2	6	E	82	ŕ	ŕ	þ.	£	ψŽ	8)	ř	ŕ	2.2	I:	_ [:	1
1/2	r e	£ £	ř B	ē.	ÉF	10 P	Ë	£ 8	žį	ξį.	DE.	*	E- [:	25	Ęį	È	<u></u>	įί
1	Ęġ	83	9	2	25	6	30	3	3	15.	\$	79	\$	Ē	.5	i p	7	ę.
	**	ŝ	8	- F	189	32	\$	3 5	88	Š	2 12	2 42	\$ 15	3 8	3 3		22	ត្ត
Prod. Avalysis, to (H. free)	-	- 5	4		- 3	•		-;	-;		9							
cis-SiX			0.00	9		<u>.</u>	3 6	201	20.5	25.0	5.5		9.7	_	c, d			٠. د
THA	37.5	# 00 # 00	7.	55.3	×.	×.	39.9	7.90	0	2.75	9.6	9.0	ر ا ا	F.	17	6.2	2.0	.0
	9.5	0.5	9.6	0.6	6	9.6	9.0	9.0	9.0	9.0			0.5		Y		_	20
Conversion, Ser	,	,			,				-			-	_	-	_		_	
CIE-CHA	12.2	12.2	, « 0 0	6.1		16.5	28.6	12.9	2.5	1.0	5:1	1.8				_		
Total Dest	2,60	4.5	-i x	× 8	?;				~ F	9 9	- C	32.3			:		_	-
THM Conver.ed To, for			:	}	!				-	?	3	y		ë	 ?:	; ;	:	
ZX										_			 	6.4	15.2	7.	4.6	31.1
Rate Constants				_									;	;	;	;		;
First Orier; sec-1	1.11	8	5.45	8.	2.5	1.0	0.88	3.6	よべ	_								1
Zero Order; eth, med-	19.27	_	38.39	_		_				89.5	37-11 (2.13	14.22	16.24	12.67	32.62	3.71	67.77
irst Order, seo	90.00	8	•	0.72	0.39	\$	0.249	397.0	0.388		0.186	- 35.	_	,			_	
Zero Order; sta, sec.	× ×	š. 	•	_					٠ <u>٠</u>	8 5		5.46		,			, ,	
				1	•		•	-			-		-	-	-	-	-	_
Zero Order			10.1	8.8					9.6	8					15.1	15.1 (10 p.s.)		11
First Order	-		- 9.5	3		•	ļ		16.0	(ata)		-						
Zero Order			5.5	5.5		-	1		15.8	15.8		1						

Bench-Scale Catalyst Evaluation Tests With Methylcyclohexane

A number of catalysts that appeared promising under micro-scale testing (MICTR) were evaluated in the bench-scale reactor. These catalysts consisted of active metals mounted on various supports. A test procedure has been described in a previous report¹⁷) which gives a measure of the effect of temperature, pressure, space velocity (i.e., contact time) and catalyst stability over a three-hour test period using a single charge of catalyst. This test involves making a series of runs at 842 and 1022°F, 10 and 50 atm, and IHSV's of 50 and 100.

The tests were done in groups with a test with the standard laboratory catalyst done in each group for reference. Different Mechanite furnace liners werequied for two groups of tests and these liners made better contact with the reactor tube wall. Presumably this better metal-to-metal contact enhanced the heat conductivity of the system and resulted in the higher MCH conversions for a given block temperature that was observed in the second and third series of runs.

The results of the tests, the conditions of each run, and the order in which the runs were made are shown in Table 25. Each catalyst was rated as to "Relative Performance". This rating was designed to show how the catalyst was performing at the end of the test, relative to the standard catalyst and quantitatively was taken as the ratio of the first order rate constant with the catalyst (k_c) to that with the standard catalyst (k_g) calculated from the MCH conversion of Run No. 7. Pased on this criteria 12 of the catalysts were superior to our standard catalyst (11 to 32% more active); one performed about like the standard catalyst; and one was considerably less active and extremely unstable and descrivated badly during the initial run at 842°F

Activation energies ranged from 10.6 (standard catalyst) to 15.5 kcal/mole. These values were calculated from the rate constants obtained from the data of Runs 1 and 2. All of the activation energies were greater than that of the standard catalyst; this suggests that the new catalysts would be even more active than the standard catalyst at temperatures above 1022°F.

The complete the property of the property of the contract of t

Table 25. DEHYDROGENATION OF METHYLCYCLOHEXANE

Evaluation of Various Catelysts

Feed: Pure MCH Reaction Time: 20 minutes

Run Munber	1	5	٤	4	2	9	1	E, ACT kcal/mole	52/2 <u>3</u>	Relativa Performance, kc/ks
3. darnteradmel about	2 118			1022	8		Î			
Pressure, atm	q	ន	30	21	91	3	OĮ.			
LHSV	8	8	8	8	8	8	100			
MCH Conversion, Sw for Catalyst No.						First	First Series			
9874-7 B)	47.4	41.4 66.0	59.8	5.69	9.8 69.2 96.1 55.7 66.9	55.7	6.90	12.6	1.80	1.00
10280-121	43.2	71.5	7.09	4.57	71.5 60.7 72.4 95.4 70.7	7.07	70.4	9711	1.03	1.11
10280-121; Muffled	41.9		51.2	72.2	72.6 51.2 72.2 97.5 71.4 71.4	4.12	4.17	15.5	1.37	1.15
10280-124A	12.5	12.5 73.7 62.2 74.4 98.1 73.0	62.2	74.47	28.1	73.0	72.8	13.4	1.17	1.21
10280-129	42.6	72.8	£.0	73.4	72.8 2.0 73.4 97.8 72.2	72.2	72.5	13.7	1.21	1.15
						Secon	Second Series	50		
7-4786	8.84		9°29	6.47	74.3 67.6 74.9 75.9 71.7 71.0	77.77	71.0	12.7	1.8	1.30
9874-73	0.84	48.0 76.2 59.3 77.4 69.6 74.9 74.4	59.3	77.4	9.69	6.47	4.47	12.0	0.95	य:1
10280-129	47.0	47.0 78.9 67.1 80.0 98.9 77.1 77.1	ó7.1	80.0	98.9	π.1	7.1	14.2	1.12	1.19
a) Standard catalyst.										(Continued)

Teble 25 (Cooki. DEHTOROGENATION OF METHYLOTOIDHERANE

Evaluation of Various Catalysts

								1 P.C.		Relative
	-1	N	>	*	~	9	. ~	7 kcal/mole	La/Le	Performance, ke/ks
						Third	Third Series			
9874-78)	£7.3	68.2	68.2 61.7 70.4	4.07	o: #	94.0 67.5	5.89	10.6	8.1	1.00
10280-1048	45.2	3.7	63.0	3.0	97.8	97.8 74.6	73.7	13.4	1.86	1.16
10280-1060	₽.9	25.9	63.2 73.6	73.6	97.3	97.3 72.3	72.8	12.4	1.17	1.12
10280-1058	42.4	68.3	68.3 61.1 69.8	69.8	93.6	93.6 67.5 67.7	67.7	12.2	1.16	0.97
						Fourth	Fourth Series			
9974-78)	42.9	₹769	8.29 4.69	72.5	93.9	93.9 68.3	68.5	11.5	1.8	1.00
10280-750	i.8.0	76.9	76.9 64.7 77.5	77.5	98.9	98.9 76.1	77.6	13.1	1.14	1.32
10280-430	8.44	24.2	74.2 64.3	5.3	97.7 73.9	23.9	23.8	12.8	1.11	1.22
10280-35E	1.94	78.6	78.6 64.7	77.1	8.0	76.4	76.3	14.6	1.27	1.28
10280-43E; 1\$ Pt on Pelleted	9.54	76.9	76.9 66.4 77.6	77.6	8.8	98.8 75.6	75.7	12.5	7.09	1.24
10280-177A	86.7b)	,	•	1	•	9	•	•	•	1

a) Standard catalyst.
b) Catalyst descrivated almost completely during Run 1.

Conventional Catalysts and Catalytic Coatings

The extensive catalyst preparation and testing program begun under this contract has continued. 18) The principal emphasis has been to develop catalysts that are more active and stable than the 1% Pt/alumins reference catalyst, for endothermic reactions such as naphthens dehydrogenation. Various attempts have been made to increase activity by promoting platinum with other metals or to better disperse platinum by various techniques. The search for active substitute metals or metal combinations for the very expensive platinum and substitute supports for alumina has continued. Various disciplines of approach to catalysts have been considered in the following catalyst preparation studies (of refs 23-30).

In addition methods of bonding thin catalytic coatings (i.e., 3-8 mils) to metal walls have been actively explored. Such active catalytic coatings are desirable since they provide a method of reducing pressure drop across a reacting heat absorbing zone. Some developments have been made earlier in this field, mainly for cracking and oxidation relations. 32)33).

A total of 556 catalysts have been prepared, or obtained from proprietary and commercial sources, and nearly all of these have been evaluated. These include metal tubes catalytically coated in various ways. The granular cutslysts have been screened for MCH dehydrogenation activ ty at 10 atm pressure, usually at LHSV 100, and at 662°, 752°, and 842°F in the micro-scale test rig (MICTR). Certain selected types of granular catalysts also have been screened for n-heptane dehydrocyclization to toluene, at LHSV 10, at 842 and 952°F, and 10 atm pressure.

A number of catalytic coatings on hypodermic tubing were acreened initially. In these runs with MCH the hypodermic tubing was placed within the standard 1/4" OD reactor tube for testing. Adequate clearance for passage of the MCH and dehydrogenation products was allowed. Later the coatings were emplaced on the interior surfaces of the 1/4" OD reactor tubes for better heat transfer during the screening with MCH. Catalysts of the same composition as the coating formulation were often tested in 10-20 mesh granular form for mechanical emvenience, for evaluation of the efficacy of the formulations.

The purpose of the screening tests is to obtain a quick comparison with the reference catalysts (9874-24 or 9874-159; 1% Pt/UOP R-8 type Algos), and to eliminate catalysts with activities too low to be of practical importance. After a favorable screening, some of the more active granular catalysts or their prototypes have been further evaluated for activity and life in the bench-scale reactor with MCH and Decalin at higher temperatures. A sketch and photographs of the MICTR are shown in Figures 87, 88, and 89, in the appendix of reference 19, along with a description of operation details.

Conventional Catalysts

Preparation

The vast majority of catalysts have been prepared by impregnation of various supports with one or more metal salt or metal complex solutions;

a) Of pages 40-42, of this report.

followed by oven drying, and reduction in situ in the MICTR prior to testing. Typically only small quantities of any particular catalyst have been prepared, i.e., a few grams to 50 grams. The amounts of metals employed are within the broad limits of 1 to 50% and most commonly within the limits of 1-10%. Since virtually all the individual metals in the periodic system that are known or can be expected to have dehydrogenating properties were studied earlier or various supports (ref 19, p 121), the principal present effort has been devoted to discovering supported bimetallic or trimetallic combinations equal to or better than supported platinum. This is particularly important in view of the rapidly increasing cost and decreasing availability of platinum during the past year, 31) and the possibility of substituting cheaper metal combinations has been emphasized.

MICTR Evaluation With MCH and n-Reptane

Methyloyclohexane Dehydrogenation

The results of the MICTR tests with various catalysts are given in detail chronologically in Tables 66, 67, 68 and 70, in the Appendix of this report. Certain of the results with MCH are summarized in the following tables which contain first order rate constant comparisons with that of the reference catalysts at 752°F (1% Pt/UOP R-3 type Al₂O₃).

Gramular catalysts consisting of substantial amounts of platinum on various supports such as alumina, silica, carbon, and magnesia are generally the most active in the MICTR tests. A number of these catalysts have been found to be very active at the higher temperatures used in the bench-scale test, and some of these have better activity-stability with both MCH and Decalin feed than the reference catalysts. The ratio of first order rate constants for some of the better catalysts vs that of the reference catalyst (9874-24) with MCH at LHSV 100 and 752°F are shown in Table 26. Included are typical granular catalysts and some of the earlier candidate catalytic coatings (in granular form). These and subsequent data are based on the conversion of MCH at constant catalyst volume, and since the catalyst densities very, different weights are charged as shown in column 4 of Table 26. Thus, laworatory catalyst 9874-7 which is 36% denser than the reference catalyst has a 26% higher rate on a constant volume basis. A second factor is variation of surface area and intribule activity per unit area. Another factor is catalyst particle size range which is usually controlled within the limits of 10-20 mesh (compare runs 285 and 284 Table 26). For the same charging weight or catalyst 10230-24A which has a relative rate of 1.28 for 10-20 mesh particles, the rate is 1.06 for 10-14 mesh particles and 1.40 for 14-20 mesh particles. Thus, diffusivity is still an important factor influencing the rate in this particle size range.

Part of this study has been devoted to improving the performance of platinum on alumina catalysts. Earlier it was pointed out that higher activity than reference catalyst results on optimizing Pt content and using higher surface area supports. Also, as pointed out previously, higher density supports tend to give higher volumetric activity.

Moderate increases in the first order rates of dehydrogenation of MCH results from using complexed platimum solutions neutralized with certain acids on impregnating the same type 1 support with 4% Pt (10280-816, 81K, and

Table 26. RELATIVE RATES OF MCH DEHYDROGENATION BY THE MORE ACTIVE CATALYSTS

Period: June, 1967 to June, 1968 Reference: 1% Pt/R-8 Algos (9874-24; kref = 1.00)

Tests in MICTR at LHSV, 10 atm pressure at 752°F, no added hydrogen

		Catalyst		
Run No.	_	Description	Wt Tested	Relative Rate Kc/Ke (752°F)
266,268,27	0 9874-7	1% Pt/Harshaw 0104 Algos	0.76 g.	1.26
288	10280-45	5% Pt/type 6 support (repeat of 9874-161B)	0.56	1.26
29 2	10280-244	4% Pt/support type 1 10-20 mesh	0.56	1.28
283	10280-241	4% Pt/support type 1 14-20 mesh	0.56	1.40
284	10280-241	4% Pt/support type 1 10-14 mesh	0.56	. 1.06
294	10289-39B	4% Pt/type 2 support	0.400	1.25
296	10280-4306)	A	0.67	1.28
298	10280-43Eb)	1% Pt/pelleted type 1 support	0.643	1.19
317	10280-3306)	4% Pt/type 2 support	0.411	1.55
351	10280-77B	2% Pt/80% type 1 support 20% type 6 binder	0.746	1.38
388	10280-83D	2% Pt/80% type 1 support 20% type 6 binder	0.702	1.23
392	10280-810	2% Pt/type 1 support	0.705	1.38
415	10280-988	2% Pt/40% type 1 support 40% type 1 support 20% type 6 binder	0.516	1.31
422	10280-104В	2% Pt/40% type 1 support 40% type 1 support 20% type 6 binder	0.609	1.57
	10280-106C	2% Pt/80% type 1 support 20% type 6 binder	0.700	1.35
	10280-121b)	4% Pt/type 1 support	0.724	1.08

a) More detailed information on individual catalyst performance will be found by referring to the appropriate run number in the Appendix Tables, and Summary Tables in the text.

b) Evaluated in bench-scale, of page 40, of this report.

(Continued)

Table 26 (Contd). RELATIVE RATES OF MCH DEHYDROCENATION BY THE FARG ACTIVE CATALYSTS

-1	L	Catalyst		Relative Rate
Run No. a)	No.	Description	Wt Tested	Kc/Ka (752°F)
526	10280-129b)	4% Pt/type 1 support (muffled at 1097°F)	0.697	1.21
527	10280-130E	<pre>## Pt/type 1 support (muffled at 1097°F)</pre>	0.672	1.37

81F compared to control 81A, c. Table 27). Mufflinga) of these catalysts in air at 1112°F prior to the usual reduction in hydrogen (at 797°F) gives various additional activity improvements in certain instances (i.e., 10280-81A, 81B, and 81D). The effect was adverse in one instance (81C) and without much effect in two other cases (81E and 81F). The higher initial rate of 81F is partly ascribable to a breakdown of part as to smaller dimensions. Generally with catalysts of the 10280-81A type, autivity declines only above 1293°F muffling in air (i.e., 1444°F). (Cf catalysts of the 143 series, runs 575-578 and 595-598, Table 63, of the Appendix.) Muffling at 1112°F causes a substantial activity decline in catalysts 9874-139 and 90, and is without effect for ref catalysts 9874-7 (cf run 545 vs 554, 548 vs 555, and 558 vs 557).

Table 27. EFFECT OF NEUTRALIZATION OF PLATINUM IMPRECIVITE BY VARIOUS ACIDS ON ICH DEHYDROCENATION ACTIVITY

Period: September-November 1967 Conditions: Same as for Table 26

Catalyst	i .	Acid Used		k _c /k _s	(752°F) ^B)
Munber 10230-	Pt, S	For Neutral- ization	Run No.	Dried	Calcined at 1112°F
81A.	140)	None	378,389	1.13	1.21
81B	ų.	ı	379,390	1.18	1.39
81C	4	2	380,391	1.30	1.03
810	4	3	381,392	1.21	1.38
81E	4	14	382,394	1.31	1.29
81F	14	5	383,395	1.49 ^{b)}	1.450)

- a) k_c = first order rate constant of experimental catalyst; k_g = first order rate constant of 1% Pt/R-3 Al₂O₃ catalyst (ref).
- b) The 10-20 mesh particles broke down to smaller sizes which would tend to increase activity somewhat.
- c) Control.

a) Muffling = heating in a muffle furnace.

Various other attempts to disperse platimum on this same support, such as chemical reduction of the complex platimum salt in situ, or longer impregnation of the support with the platimum complex, gave no improvement in activity (of 10280-1437 and 176A, resp; runs 601 and 624 in Table 68, of the Appendix).

A sumber of older Pt/type 1 support catalysts have been retested for comparison with similar more recently prepared catalysts which appeared less active under present test conditions. Generally, their first order rate constants were lower relative to the reference catalyst, than originally found. This probably results in part from removal of fines. less than 20 mesh which is now routinely done. Previously it was shown that the activity increases substantially with decreasing particle size. The results are summarized in Table 28 and detailed in Table 68, of the Appendix.

Table 28. RELATIVE MCH DEHTROGENATION RATES OF SEVERAL OLDER SUPPORTED Pt CATALYSTS

Conditions: 1K5V 100, 10 atm pressure, no added hydrogen 0.9 ml 10-20 mesh catalysts diluted with quartz to 2.0 ml (cetalysts prereduced at 797°F)

Run No.	Catalyst No. 9874-	Catalyst Description	rc/kst)	Run No.	k _c /k _s (Older Data)
555	199A	4% Pte)/type 1 supportb) 4% Pt/type 1 supportc)d 4% Pt/type 1 supportc)d 4% Pt/type 1 supportc)e 4% Pt/type 1 supportc)e)	1.09	205	1.09
570	199C		1.29	208	1.44
567	199D		1.17	207	1.32
568	200B		1.08	212	1.29
569	200D		1.32	214	1.65

a) Impregnate acidified.

b) Support used as received.

c) Support muffled at 932°F before impregnation.

d) Catalyst muffled at 1095°F in air before testing.

c) Different than used for 9874-199 catalyst series.

See footnote (ε) Teble 27.

A highly active catalyst results at 4% Pt loading of a new spherical type 1 support of high density (0.76) and high surface area (285 m^2/g) (of catalysts 10280-91A and 91B; Table 66 of the Appendix). A large quantity)436 ml) of a catalyst of the 91B type has been made for evaluation in the FSSTR (10280-113).

A number of additional metals on various supports were also evaluated; and of these only metals A (previously noted) on type 1 or 2 supports, metal B on type 2 support, and metal D on type 1 support appear

a) This occurs in some cases by decrepitation resulting from contact with the impregnate solution.

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proxising (of Table 29). These have been studied further in various binetallic and trimetallic combinations on various supports (see later section).

Table 29. POTATIVE MCH DEHYDROGENATION RATES WITH VARIOUS ST. SE MELALS ON SEVERAL SUI PORTS

		atalyst Des	eription	
Rum No.	No. 10280-	Metal Type	Support Type	Ic/Ka (752°F)a)
32 5	140 A	10% 🛦	2	0.84
508	119E	1\$ A	1	0.77
671	185E	4 % B	5	0.87
337	66A	4 5 B	?	0.46
425	1027	5≸ C	1	0-0
661	172	4% D	1	∍ .82
486	111B	20% E	1	9-12
485	1114	20% F	1	0.35
290	3 6	5 % G	1	0.0
349	66в	5% G	2	0.0
299	42	5\$ N	1	0.0

a) See footnote a), Table 27.

Bimetallics

Various promoters of activity for platinum on type 1 or 2 supports have been evaluated in the MICTR. Many of these are listed in Table 30 with their relative rates at 752°F with MCH at LHSV 100. These promoters are either without beneficial effect or are deleterious to platinum activity.

Many supported bimetallics (not containing platinum) have been examined and a few of these have shown some promise. Data for one of these systems is shown in (Table 51). One of the supported (type 1) metals is completely inactive and the other supported metal alone has activity up to 752°F but becomes inactive at 842°F (10280-102F and 66A, resp). Over a wide range of compositions these two metals cooperate with a broad maximum of activity at 662-842°F. The highest rate, however, is only 80% of that of the reference catalyst.

Trimetallics

Many trimetallies on type I support have been evaluated in the MICTR with MCH; one of these metals was platinum. None of the metal combinations used in the explorative phase promoted platinum activity, and a number were deleterious (of Table 32).

Some of the more promising metals were evaluated also in bimetallic or trimetallic combinations in different proportions on type 1 support (cf

Table 30. RELATIVE MCH DEHYDROGENATION RATES WITH SUPPORTED VARIABLE COMPOSITION BIMETALLICS

Period: March-May, 1963
Conditions: LHSV 100, 10 atm pressure, no added hydrogen. 0.9 ml 10-20 mesh catelyst diluted with quartz to 2.0 ml (catalysts reduced in situ).

Rum No.	Catalyst No. 10286-	Pt	Bimetellic Promoter	Kc/Kn(752°F)
	100 20200			357347 152.3V
		177	e 2 Support	
667	185A	4%	O≱ B(Control)	1.0
668	165B	3%	1% B	0.96
669	185C	2%	2% B	0.97
670	185D	15	3% B	0.84
671	185E	0	4% B	0.87
		Typ	e 1 Support	
624	176A	4%	0	1.14
636	157B	3\$	1 % B	0.87
635	157A	1%	3% B	0.86
337	66A	o	4 % B	0.40
369	79B	1%	0 (Control)	1.00
370	79C	1%	1% C	0.35
371	aer	1%	1\$ L	0.49
3T2	79E	1%	1% M	0.93
315	42A	1%	1% H	0.44
373	79 F	1%	1\$ N	0.60
375	80C	1%	15 F	0.68
576	80B	1%	1% E	0.68

Table 33). Once again none of these combinations produced a more active catalyst their platinum alone and some were deleterious to activity.

Table 31. RELATIVE MEN DEHYDROGENATION RATES WITH VARIABLE CONFOSITION BIFETALLICS ON A TYPE I SUFFORT

Poriod: September-November, 1967 Conditions: LHSV 100, 10 atm pressure,

no added hydrogen 0.9 ml, 10-20 mesh catalyst diluted with quartz to 2.0 ml (catalyst reduced in situl-

Run	Catalyst	Bimet	allic	
No.	No. 10280-	% C	9 B	$K_{\rm C}/K_{\rm R}$
425	102F	5	0	C
426	102A	2	4	0.74
427	102B	5	5	0.78
428	102 C	10	4	0.69
446	109B	10	2	0.74
429	102D	5	2	0.69
430	103E	5	6	0.67
337	66 h	Ó	Ĺ.	0.46

Table 32. RELATIVE MCH DEHYDROGENATION RATES WITH VARIABLE COMPOSITION TRIMETALLICS ON A TYPE 1 SUPPORT

Run	Catalyst	Tr	imetall		
No.	No. 10280-	<u> </u>	2 (5)	\$ (3)	Kc/Ka (752°F)
624	176A	4% Pt	0	0	1.14
657	163A	3% Pt	15 E	1% D	0.88
658	153B	35 Pt	1% F	1% D	0.91
659 660 664 665 639	163C	3% Pt	19. A	15 D	0.97
660	163D	3% Pt	1% G	1% D	1.04
664	163E	3% Pt	15 H	1% D	0.91
665	163F .	3% Pt	1% I	1% D	0.72
639	159 C	1% Pt	1% B	34 D	بلو.٥
640	1614	1% Ft	1% E	3% D	0.75
641	161B	1% Pt	1% F	3% D	0.79
645	191D	1% Pt	1% G	3% D	0.89
646 647	161C	1% Pt	1% H	3% D	0.78
647	161F	1% Pt	1% I `	3% D	0.69

s) Period: March-Muy, 1968, Conditions as in Table 31.

Dehydrogenation of n-Heptane

A cross-section of various catalyst types have been screened for dehydrocyclization of n-heptane to toluene, at LESV 10, 10 atm pressure, and at 842 and 932°F, without added hydrogen. At complete conversion the combined sensible and reaction heats expected at 932°F would be about 955 Btu/lb for cracking and 1774 Btu/lb for dehydrocyclization. The results of these tests are given in Table 70 of the Appendix, with catalyst 9874-24 used as reference.

Table 33. RELATIVE MCH DEHYDROGENATION RATES WITH VARIOUS AMOUNTS OF SUPPORTED BIMETALLICS AND TRIMETALLICS

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Conditions: LHSV 100, 10 atm pressure, no added hydrogen, 0.9 ml 10-20 mesh catalysts diluted with quarts to 2.0 ml (catalysts prereduced at 797°F). temperature = 752°F

Catalyst	Fere	ent i	y Yt	Run	Ko/K a)b)	Run	Ko/Ka*)0)
No. 10280-	Pt	A	P	No.	(752**)	No.	(752*)
119A	1	U	U	502	1.05		
121	4	0	0	808	1.13	•	j -
1198	0	1	0	508	0.77	-	
11938	1	ĺ	0	508	0.97	•	
119C	1	2	0	503	1.06	-	
1190	1	3	0	507	0.95	•	
1190	1 2	1	0	512	0.92	541	1.03
1:05	2	1 2	0	515	3.06	539	1.14
119H	3	1	101	513	1.14	538	1.25
1191	3	3	0	516	1.09	840	1.03
120D	0	0	1	520	0.13	-	-
120A	1	0	lıl	517	0.70	. •	-
120B	1	0	2	518	0.63	_	•
120C	2	0	lзl	519	0.60	-	
120g	2	0	lil	521	0.79		1 -
1209	3	٥	1	522	0.86	•	i -
120H	3	0	2	524	0.88	•	
1200	3	0	3	523	0.74	•	
122A	1	1	1 1	532	0.94	•	
1227	1	1	3	535	0.84	•	
122D	1	3	3	533	0.93	-	1 -
122H	3	1	1 1	535	1.19	-] -
122E	3	3	lı	534	1.11	•	-
1221	3	3	2	537	0.93	•	•
1228	3	3	3	520	1.00	•	
122C	3	انا	3 1	830	1.01		-

- a) K_c = first order rate constant of experimental catalyst K, a first order rate constant or (9874-139) (15 Pt/AlgO3 R-8 type). # first order rate constant of reference catalyst
- b) 256°F dried before reduction.
- c) Muffled at 1112°C in air before reduction.

Many of the catalysts were completely inactive under these conditions and others formed substantial amounts of cracked products as well as toluene at the highest temperature; little or no hexenes were formed. In several instances almost complete conversion to cracked products occurred (i.e., 6749-33B, 10280-111A, 111B, and 117C; Table 70 of the Appendix). In view of the low allowable space velocity (10) as compared to MCH dehydrogenation (100-200), as well as the undesirable nature of the products, none of the catalysts tested under these conditions appears promising for heat sink usage. In cases where extensive cracking occurs carbon formation would be expected to limit catalyst life. However, a very active cracking catalyst might be useful as a dispersed phase catalyst (especially if some dehydrocyclization also occurred).

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Catalytic Coatings

Preparation and Granular Evaluation With MCH

Preliminary experiments were made by coating stainless steel with thin layers of catalyst supports. Stainless steel strips (1" x 2") were first sand-blasted and then degressed with scetons, prior to coating most of the trials were made by starting with wet paste mixtures prepared from type 1 supports and various binding materials. The pastes were emplaced by ameaning with a spatula on the strips and in some cases a scalp massage vibrator was used to smooth the thixotropic ocating. After drying in air and at 284°F, strips with coatings having good adhesion were heated in a muffle furnace at 932°F or lower temperature for a further check. Coatings were about 20 mile in thickness. Coating cardidates were also placed on the exterior of roughened hypotermic tubing, 8) stainless steel screen, or made as granular supports which after platinizing were tested in the MICTR. Evaluation of platinized granular formulations is convenient and useful to determine the catalytic efficacy of the coatings. The evaluation of mechanical properties of the earlier coatings is shown in Table 69 of the Appendix which gives the number of granular or tube catalysts derived therefrom. Good adhesion was obtained with an 80% mixture of finely ground fibrous type 1 support and particulate type 1 supports (1:1) and 20% type 6 binder (formulation I), or with a particulate type 1 supports and type 6 binder (4:1) (formulation II). Supports of this type (after platinizing) are very active for MCH dehydrogenation (of hims 415, 417 and 492, in Table 35, next section).

A large number of these catalysts have shown activity exceeding that of the reference catalyst and equaling that of the best previously prepared granular catalysts on the same volume or weight charging basis (i.e., 77A, 77B, 106C, 75E, 9B, 89B, 98C, 104B, 92A, 92B, 92C and 98D). The most satisfactory non-shrinking and adherent formulations are still those prepared from fibrous and particulate type 1 supports and type 6 binder-Adherence to sand blasted stainless steel strips is responsity good, but further improvement in this respect is desirable. Attempts were made to raise activity per unit volume by using a high density (low surface area) type 1 particulate support in the formulation, but little is gained in activity per unit volume in raising the density beyond about 0.7. The best result has been obtained with catalyst coating formulation 10280-980 with 3% Pt loading (of Table 34). Impregnation of coating materials with another metal, that is almost as active and selective as platinum at high MCH conversion, led to similar results obtained earlier with this metal on conventional supports which were side reactions leading to benzens and cracked products (catalysts 10280-104C and 106D).

a) 8" long x 0.11 dia. with 0.012" wall thickness, crimped at ends.

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Table 34. RELATIVE MOH DEATDROGENATION RATES WITH VARIOUS CANDIDATE CATALYTIC COATINGS (CRANITAR)

September - November, 1967 LHSV 100, 10 atm pressure, no added hydrogen 0.9 at 10-20 mesh catalysts diluted with quarts to 2.0 ml (catalyst reduced in situ) Conditions: Perfod:

Bulk k _c /k _s c) Density (752°F)	0.36 1.10	0.54 1.25 0.85 1.38 0.77 1.35	0.47 1.00 0.50 1.46 0.56 1.27 0.50 1.05 0.77 1.08	0.82 0.54 0.81 1.11 0.56 1.11
2.0	311	492f) 351 623	# 2 # 2 # 2	× × × ×
Support Adhesion Te Metai Strip	Food	Food -	figure of Poor	60 dd dd 60 dd
Support Description	80% type I support - 20% type 6 binder	<pre>405 type i support (i), 405 type i support (2) - 205 bincer type 6 805s) type i support - 205 binder type 6 805s) type i support - 205 binder type 6</pre>	41% type i support (1), 41% type is) support (2) - 16% binder type 6 41% type i support (1), 41% type is) support (2) - 16% binder type 6 41% type i support (1), 41% type is) support (2) - 16% binder type 6 41% type i support (1), 41% type is) support (2) - 16% binder type 6 80% type i support - 20% binder type 6 80% type i support - 20% binder type i	805 type 1b)e support - 205 binder type 6 805 type 1b)e support - 205 binder type 6 405 type I support (1), 405 type 1b)e) support - 205 binder type 6 405 type I support (1), 405 type 1b)e) support - 205 binder type 6
14 E	7	222	- 22444	- 22 - 22
Catalyst No. 10280-	63.A	77.8 778 tagc ^{e)})	71.7 71.7 72.6 73.8 53.8 58.8 58.8	83.4 838. 928.

kc " first order rate constant of experimental catalysts. ks = first order rate constant of 15 Pt/R-8 Ai₂O₃ citalyst (mf.). As received. コゴマ

Prepared for bench scale test evaluation. Surface area 8.7 s2/gs. めるむ

Repeat of Run 150, c.f. Table 66 of the Appendix.

Table 34 (Coutd). Relative was desidence alternated vite various candidate catalytic coalings (Grandlar)

894 i 405 type [support (!), 838°5'u) 2 405 type [support (!), 965°5'd) 2 405 type support (!), 966°5'd) 2 405 type support (!), 966°5'd 3 405 type support (!),		Betai Strip	- d	WE'RE CY	(752 T)
2 405 type support (1), 2 405 type support (1), 2 405 type support (1), 3 405 type support (1),	to type !	be. Sa	38 38	0.62	1.15 1.37
2 (40) type I support (!), 3 (40) type I support (!),	40° type 1 support ⁸ (9) (2) - 20° 40° type 1 support ⁸ (6) (2) - 20°		= = =	0.67	\$ R
•	40% type i supportably (pallated) 40% type i supportably (2) - 20%	, ,	=======================================	0.62	
4 40% type I support (!), 2 40% type ! support (!).	40, type 1	Legs Legs	= 2	0.63	1.16
1 27% type I support (!).	53/ type I supportb) (2) - 20/ type 6		SE\$	0.75	22.
Z Z/F type support (1),	$M_{\rm c}$ type I supported (2) - 305 ty 67; type I supported (2) - 305	5000 der 5000	축 축	0.73 2.73	27.
2 13% type 1 support (1),	67; type 1 supporta(b) (2)		503	0.81	92"
[80% type i supportels]	M. type	Poor	387	57.0	90°1
	3 - 206 type 6 bloder	- P	8	8.0 E.0	1.23
1 852 type 1 supportalb)	155 type	Fair	\$	6.83	1:24
2 85% type (support*)0)) - iSμ type 6 binder	<u> </u>	ğ	0.84	:.25

Ball allied.

Surface area 11.3 a2/ga.

Impregnate carbonated.

Lower density because platinum impregnating solution maked during mining support ingredients. Prepared for bench scale test evaluation. Catalyst calcined at 1112%.

is received.

Further experimentation has been carried out with the adherent synthetic fibrous-particulate type 1 support mixture with the type 6 binder (formulation I; 40:40:20). Satisfactory acherence and activity (when platinumized) result when particulate type supports ranging from 7 to -278 m2/g are used; the latter giving an overall surface area of -250 m2/g. The use of a highly alkaline type 17 binder leads to poor metal adherence and poor activity on platinization (of run 544 and 546 of Table 67 of the Appendix). Various treatments of formulation I type support, or catalysts derived therefrom, have somewhat different effects than the corresponding treatments on conventional supported catalysts where these treatments resulted in improved activity. Acid neutralization of the impregnate decreases the relative dehydrogenation rate i'rom 1.12 to 0.81 (catalysts 133A vs 135B, Table 35). A lower relative rate (0.98 vs 1.12) is obtained on diving the support at 259°F instead of muffling at 1112°F, before metal impregnation (of catalysts 1330 vs 133A, Table 35). A slightly decreased relative rate (1.06 vs 1.12) results from muffling the finished catalyst in air at 1112°F (of catalysts 133D vs 133A, Table 35).

Substitution of 40% of type 2, 10, or 19 supports (formulations III, IV, or V) for 40% particulate support 1 (formula I) leeds to satisfactory adhesion to stainless steel particularly if first coated with binder 18, as described below. Formulation III was designed to raise the surface area of various formulations I from the range of 120-250 m²/gm to ca 500 m²/gm (of granular catalysts 117A and 117B, of Appendix, Table 67). Granular supports of these types give catalysts with activity equal to or greater than the reference catalyst.

Addition of 12% of an oxidizable particulate metal dust (type 15) to formulation I gives some improvement in adhesion and a satisfactory granular catalyst support (of catalysts 131A and 131B, of Appendix, Table 67). This metal in finely divided flake form was found to be highly reactive with water in the formulation, with resultant frothing and heating up to form a highly porous friable support which was not usable.

Substitution of a fibrous mineral (type 16) for the fibrous synthetic type 1 support in formulation I has been studied. The former is readily available while the latter is no longer manufactured. One finely ground sample led to a formulation (40% type 1, 40% type 16, 20% type 6 binder) that when platinized had satisfactory activity (of catalyst 140A, Table 35). This formulation (VI) is less thirotropic when wet and gives poorer metal adhesion after muffling than formulation I. Two other samples of type 16 material give formulations which crack and strip from metal even on air drying. Formulations of this type can probably be improved as to adhesion.

There are various problems associated with binding a thin catalytic coating onto metal so firmly that it adheres well at variable elevated temperatures and survives temperature cycling. First, there is a difference in two dimensional thermal expension between the coating (an insulator) and the metal which on heating induces mechanical strain on the coating. Second, a tight bond is necessary between the catalytic coating and the metal wall so that coating should have some elasticity to prevent "stripping" and thus allow efficient heat exchange between the metal wall and the reacting fluid. Third, the coating itself must have good mechanical strength, so it remains in situ for a long period of time in use. An upper limit is set upon the strength of

Table 35. RELATIVE MCH DEHYDROGENATION RATES WITH VARIOUS CANDIDATE CATALYTIC COATINGS (GRANULAR)

Conditions: LHSV 100, IC atm pressure, no edded hydrogen, 0.9 ml 10-20 mesh catalysts diluted with quartz to 2.0 ml (catalysts prereduced at 797°F) (cf test data Table 67 of the Appendix). T w 752°F.

Catalyst No.	Pt.	Support Description	Run No.	Bulk Density	k _C /k _s a)
9874-139	ī	UOP R-8 type Al ₃ O ₃ (ref)	543, 559	0.47	1,00
10280-1334	3	40% type support (- 40% type suprort (2) - 20% type binder ^b)	552	0.53	1.12
² -1338	30)	40% type I support (I) = 40% type I support (2) = 20% type 6 binder ^b)	563	0.53	0.81
" -133C	3	40% type I support (1) - 40% type I support (2) - 20% type 6 binder ^{c)}	564	0.55	0.38
-1320	3	10280-133C muffled at 1112字b)	551	0.52	1.06
* - 77X	2	40% type support (1) - 40% type support (2)*) - 20% type 6 binder	492	0.56	1.24
a -1317	3	39% type i support (1) - 39% type i support (2) - 12 exidizable setal - 10% type i binder [©])	553	0.55	1.10
ª -1316	3	39/ type support (1) = 39/ type support (2) = 12 exidizable sets! = 10/ type binder ^d)	560	0.52	1.10
3 -117Å	2	40% type 1 support - 40% type 2 support - 20% type 6 binder	487	0.37	1.19
• -1178	4	40/ type I support - 40/ type 2 support - 20/ type 6 binder	488	0.37	1.12
-14CV	3	40% type 1 support - 40% type 16 support - 20% type 6 binder	\$56	0.33	1.03

- a) kc = first order rate constant of experimental catalyst.
 - k. m first order rate constant of reference catalyst 9874-139.
- b) 1112°F muffled support used for impregnation.
- c) 259°F dried support used for facegnation.
- d) Acid neutralized impregnate.
- e) Different type than used for catalyst 10260-133 series.

the catalytic coating since it must have a substantial pore volume and surface erea to perform properly and therefore cannot be as hard or bonded as tightly as a sintered nemporous ceramic coating. Some of these problems can be resolved mechanically but certain of them are inherent.

The above described tests were carried out with sheared 1- x 2-instrips of stainless steel and consequently had cambered shapes. This causes unequal stress in the longitudal direction on muffling in air and therefore an uneven strain on the estalytic coating. Mild steel strips have been obtained

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for coating studies which have been band sawed to shape to avoid this problem. This metal oxidizes more readily on muffling than stainless steel but this would not occur in a reducing atmosphere.

An improvement has been made in binding formulation I type material to steel surfaces. The cleaned surface is given three applications of 5% aqueous type 18 binder. Each coating is air dried, and finally the triple coat is air dried and muffled at 752°F for 1 hour. This apparently forms a glass like surface bond to the metal and in turn forms a good bond to the formulation. Also it reduces oxidation on muffling in air. All 1/4" OD resorter tubes coated with catalyst coatings were first coated with type 18 binder (of next section, Table 36). Pt coated binder is invetive (of run 592, in the Appendix, Table 67).

Various formulations were coated onto the walls of 1/4" OD tubing, the tubing wall thickness was usually 0.028". A fairly well controlled thickness was obtained by first partly filling the upper end of a tube with the thixotropic formulation and then drawing a Teflon plunger through (both ends tapered) with the desired diameter. The tubes were dried at 126°C, the excess coating drilled cut with a No. 10 numbered drill to the desired coating length, and then muffled at 732°F. This was followed by filling the tube with the platinum or other impregnating solution, followed by drying and reduction in situ. Thicknesses were varied from ca 4 to 15 mils, and were usually around 7-1/2 to 8-1/2 mils. An effort was made to keep the platinum content in the 2-5% range. This could be determined accurately only by drilling out a catalyst coating and having the platinum content analyzed. The not gain in weight on platinizing is a very insensitive measure of platinum content because of the considerable tube weight (~58 pm) as compared to catalyst weight (0.1-1.0 pm) of which the Pt content is only 2-5%.

Coating Evaluation With MCH

The results obtained with various catalytically coated 1/4" tubes on MICTR testing with MCH are summarized in the following Table 36, and given in more detail in Table 68 of the Appendix.

At constant pump rate (90 ml/hr), corresponding to an MCH LHEV of 100 with conventional granular particles, 8" coated tubes 1 and 2 give higher relative rates than the reference catalyst, particularly on the same catalyst weight basis (cf last column of Table 36). As with all the other coated tubes described in Table 36 higher rates are obtained if quarts packing is used to create turbulence. An unpacked tube has an estimated Raynolds number of ca 350, at 752°F (i.e., lamellar flow range). In part, higher activity results from a thin longer catalyst zone (8 vs usual 4") which allows better heat exchange so the catalyst operates closer to the block temperatures than the shorter bed granular catalysts. Activity increase also has been shown with identical amounts of ref catalyst 9874-24 by doubling the diluted bed length from 4 to 8" which gave an apparent rate increase of 46%, and with the other ref catalyst (9874-159) an increase of 29% (cf runs 333 vs 334 and 603 vs 604, Appendix Tables 66 and 68). Also, finer particle size (synonymous with a thin layer) was shown earlier to give an increase in rat. (cf Table 36).

Three tubes (3,4, and 5) coated with different weights and thicknesses of platinized formulation 1 gave about the same high rates without quartz

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packing and higher and equivalent rates with quartz packing. The highest rate of this series vs that of an equivalent weight of ref catalyst was with the tube with the thinnest coating (tube 5, quartz filled, run 610).

A thin coated tube (10) with 4" bed length gave a high relative weight rate (7.32) when quartz packed. Annular tubes 0.125" dia or 0.177" dia were centered in the catalyst tube to create turbulence. However, the increase in turbulence might be more than counterbalanced by the decrease in contact time. The relative rates decreased, and a recheck of the quartz filled coated tube showed the relative activity had declined during this series of touts, probably because of mechanical injury and loss occurring while making the physical arrangements. The experiment will be repeated.

The highest relative weight rate was obtained with a quartz filled tube (7) coated with formulation III support (of run 600). High rates were also obtained with formulation IV coated tubes, and repeat runs with ani without quartz gave about the same results.

Another metal (A) performed better, relative to platimum, on a coated tube than on gramular support at high conversion, however, come benzene was formed in addition to toluene. Experience with the bench-scale tests indicates a gradual loss of activity. A second metal (D) performed fairly well but was not a serious competitor (run 517, Appendix, Table 68).

These overall results show considerable promise for the use of this catalyst coating for the proposal andothermic catalytic heat exchanger and this effort will be continued.

A few experiments were carried out with an 80 mesh Pt-Ir (90:10) screen which weighed 0.85 gm. This was loaded into a $1/4^{\circ}$ CD reactor tube and MCH passed thru it at a rate of 45 ml/hr. The screen was inactive, in this form and also when coated with 0.25 gm of Pt black (of runs 327 and 330, Table 66 of the Appendix).

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Table 56. EVALUATION OF MCH DEHYDROGENATION WITH VARIOUS CATALYTICALLY COATED TUBES WITH DIFFERENT PHYSICAL ARRANGEMENTS

Pt impregnated unless otherwise noted Conditions: same as for Table 35, constant pump rate (90 ml/hr)

Rus	Catalinat			Conting		Querte	м,	Coay 1	Tche ve		
No.	Cotalyst No. 10280.	Tube No.	Gi ama	Thickness,	Longth	in Tube	612	752	9-5-A	r _c /t	Neintive Sate, we 0,42 g := f catalys
582		1	0.239	7.5	-	-	26	71	81	1.89	3.38
585	151	<u> </u>	(Form 1)	1.5	"	x	37	76	97	2.19	3.92
584		(مر	0.106	11.5	8-		29	61	77	2.45	1.12
585	15°	[(Fore 1)	11.5] =	y	41	75	96	5.11	2.52
50 0	158		1.00	15	g-	T	>6	65	78	1.54	0.65
587	170	,	(Fore 1)	15	-	*	30	71	3 2	1.98	0.83
500			0.663	9.4	8-		29	59	77	2.57	0.06
589	158	·	(Form 1)	8-1/2		×	31	72	95	1.95	1.26
590	158		0.291	-4	8-		27	44	85	1.59	2.38
591	190	,	(Form 1)	l "] **	×	41	76	93	2,38	9.44
615	170	,,	0.228	-6	8-		31	67	85	2.77	5.26
610	170	11	(Form 1)	•		×	32	81	98	2.02	3.19
627			0.0009				22	46	60	0.98	4.64
626	170	10	(Form 1)	•		X	26	62	61	1.55	7.32
634	170	مد	0.0.09	4		b 1	25	47	62	0.97	4.64
651	170		(fore 1)	-		•)	17	40	60	0.77	3.69
676	194	20	0.0509	-46		•}	Tr.	25	32	0.44	8.38
6754)	170		(Poru 1)		, "	×	26	4.8	72	100	4.8
599	165	7	0.073				27	65	83	1.60	7 9.4
600	107	(Porm III)	0.075	7.5	9"	×	29	74	95	2.04	11.9
67.9	1760	15 (Form IV)	0.527	8.5	7.73	×	وز	76	96	2.17	2.75
650	178C	1h (Form IV)	0.167	8.5	4.25	x	31	77	96	2,2)	5.70
572 573	176ef) 176cs)	15 14	727.0 721.0	8.5 6.5	7.75 6.25	X	28 28	83	96 93	2.17	2.62 4.53
210 220	173	6 (Form 1)	2.265 ^h)	7.5	7.5	x	25 37	57 79	68 96	1.28 £.40	3.76 1.05
527	173	y (Form 1)	0.3351)	7.5	7.5	×	æ	ю	64	1.05	L.))

a) Wall thickness 0.032", all others 0.020",
b) Home above cetalyst (illed with quarts.
c) 0.125" diameter annular tube contend inside coated reactor tube.
d) Hechack of run 656 on original catalyst activity.
c) 0.177" diameter annular tube centered inside 1/b= 0D coated reactor tube.
f) Hepest of run 651.
b) Impregnated with metal A.
l) Impregnated with metal B.

Nonconventional Catalyst Systems

Homogeneous and Dispersed Catalysis

As discussed in an earlier report¹⁹⁾ at area of catalyst modifications which rould be extremely useful to heat sink applications involves providing a catalyst dispersed in either a discrete or molecular state, which could be introduced with the fuel in the heat exchange zone of an engine, and then proceed to the condustion chamber through the nozzle. Here we are investigating, in a preliminary way, possible fuel soluble or fuel dispersible catalytic materials, and testing them for their effect on the MCH dehydrogenation reaction.

Most of the preliminary work to date has involved the simple process of adding MCH and the catalyst in the desired amounts to a stirred autoclave and then observing the pressure rise during the vaporization and reaction. Analysis were by GLC. Several materials have shown catalytic activity at 1 percent metal (basis weight of MCH) at 800-1000°. Nearly all tests have been run in the virtual absence of oxygen.

Results from the autoclavs tests have been tabulated in <u>Table 37</u>. Catalysts used have cone from both commercial sources and lab preps. Size encouraging results have been obtained in a few cases, and actually most of the compounds tested have produced at least some activity compared to the neat MCH base case.

One material (Sums 126-129, 155-155) showed remarkable activity, and in Runs 126 and 127, with good specificity to toluene and benzens. He pressure developed to high levels for these runs also. Unfortunately, the activity of this catalyst has proven to be elusive and nonreproducible. The other runs shown for this catalyst represent efforts to find the cause for this non-reproducibility, by varying temperature and oxygen content of the blanketing gas. Run 150 combined the catalyst of interest with the catalyst tested in run 125 to check out the possibility of synergism by contamination with the previous catalyst. The activity state of the autoclave surface has also been considered as a possible factor in this enigmatic behavior, but has not been investigated. So far, all efforts have failed to reproduce the excellent results of Runs 126 and 127.

Two compounds (Runs 124 and 125) gave about 50 percent total conversions, but these appear to act mainly as cracking catalysts, since most of the products were lower molecular weights than MCH. Another active catalyst which appears to fit in the cracking category is that of Runs 145, 146, 156, and 158.

Runs 118 and 119 are also of interest because of their relatively high total conversions to dehydrogenation products (ca 25 percent toluene, bensene, and methylcylohexenes).

The degree of activity displayed by these compounds is ensouraging, although most are less soluble than would be desired. The results may, in some instances, point the way to modifications in the organic ligands which will improve this and other properties. We have also been doing some work on the concept of using small amounts of special solvents (solubilizing agents)

Table 37. DISPERSED PHASE DEHYDROGENATION OF MCH

Catalyst additions = 1.0 w metal, basis weight of MCH

	Campos		č.	nseralar & Pr	alurul!		Polec	Dy Frankant of No Hua	Streety	toponer time	, trum
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97	j .	b.3		0.1	0.7	4.6	173	17.7	761	0.7	16
£A 09	,	0.3	0.1	0.1 0.1	6.4 0.3	0.6	1 <i>P</i> 0 150	167	809 164	#.0 3.0	7.3
*	1 , 1	1.0		0.1	, ,	4.0	146	167	16-4	1.5	6.4
95	6	0.5	0.7	6.3	. •	1.7	:75	147	4.4	2.7	7.4
46 9 7		0.5	Trees	G. ♥ 3	6.5	3.4	157	167	PM AA	1.0 3.9	7.3 6.3
. ₹1 ₩	10	1.3	0.1	a.5	D.#	7.1	3.40	-37	, T	2.7	7.8
100	10	0.4	Treve	0.1	€.3	1.9	111	147	5:5	3,6	7.0
101	ע	0.1	-	30	0.0	3.7	1.4	147	/09	0, y	6.8
100 103	15	0.1	:	0.6 1.0	3.2	0.9	116 215	747 741	A35 A34	2.9 5.6	5.8 7.9
104	15	0.1	:	6.3	0.5	G.6	11'	11.7	Lag.	5.5	6.9
105	16	0.1	(·	0.8	G. <i>3</i>	0.5	120	147	T#	1.2	4.9
106	17	0.1	:	0.1	0.8 3.8	0.6	1.0	167	Roo	2.7	7.2 6.2
10† 10 0	19	0.1	0.8	0.1	3.2	1.1	Tr. P1	147	863 AQ2	5.1 5.9	7.8
10)	20	0.4	0.1	0.5	0.5	1.4	•	147	A 01	5.3	8.2
110	[#a	0.1	Trees	0.1	6.3	0.6	מ	147	60)	6.5	7.4
111	n	0.1	Trans	71666 7.8	6.3 6.5	12.0	155	347 347	649 671	5.0 4.75	7 b 6.6
112 113	2)	5.0	1rane	4.5	6.9	16.4	117	147	€.66	6,0	B.1
110	n	3.8		9.0	4.9	13.7	125	347	M 5	1.2	8.5
115	1 36	7.4		3.0	9.0	15.8	143	114	* #	4.5	7.0
95 317	1 1	9.3	4.6	3.3	9.6	36.5	141	367 167	N1:	0.53 5.7	0.0 7.4
110	**	16.2	36	1.5	2.1	23.2	167	147	900	·	6.9
119	29	17.0	5.6	C.9	2.6	26.1	158	167	yxx0	5.5	2.6
750) 3 0	2.3	0,5 2,9¢}	9.0 9.30	6.9 51.8 ^{e)}	32.3	N	167	900	1.0	7.1 6.8
121	и я	0.6	3.9"	2.6	8.6	11.9	25-2	347 347	200 201	6.5 6.5	5.8
u)	1 2	4.5	13	5.4	3,.4	25.1	109	147	900	3.6	7.0
334	×	6.6	4.1	6.6	36,4	49.8	D>	147	y00	\$.1	7.2
127 186) X)	3.8	3.1 47	4.5 2.7	39.6 5.1)2.4 (36.8 ∣	1MB 500	167	910	1.0 6.0	9.8 6.0
437	×	5.1	3.0	0.8	1.5	9.2	136	964	700	4.0	6.2
155	1 × 1	13.3	8.5	1.1	3.0	19.9	136	(1.515)	900	4.5	8.5
194	¥	4.0	۵.0	1.7	1.7	10.7	103	(Alr)	900	4.3	6.6
127 136) % %	6.A	90	1.1 0.5	2.0 1.2	98.3 2.9	532 15	347 247	497 661	6.0 6.0	8.0 6.9
124	x	6.6	3.9	1.1	13.7	25.5	121	217	#46	6.0	1.3
150	¥, ₩	2.1	1.0	1.1	3.1	7.6	121	147	8-4	7.6	8.3
131	1 12	1.2	0.6	3.4 0.2	4.8 4.7) . 11.2	11	1.6	900	3.5	6.0 7.6
136 137	13	5.7 6.0	0.0	1.4	2.0	9.7	89	1.6 1.6)OC Bys	9.3 9.6	7.8
136	1 44	4.7	0.4	1.4	3.6	0.1	**	1.6	900	5.0	7.0
199	159	1.6	6.2	1.1	∌,a	5.1	77	1.6	900	3.4	A.0
7#7 7#0	4,7	1.0	0.4	0.7 1.3	3.5 4.0	9.6	#44)	1.6 1.6	901 903	5.7 5.7	7.2 6.2
144	ŭ	0.6	0.2	0.7	4.5	6.0	111	1.6	90t	5.5	8.0
296	.,	3.7	2.2	U.6	39.4	35.5	145	3.6	7077	2.0	3.1
140	***	3.6	1.8 3.8	0.5	27.6 16.6	96.7 19.3	195 87.5	1.6	:01 100k	6.9 6.2	5.7 8.0
144	37	1.0	1russ	3.6	Na.0	9.5	59	1.6	7.6	6.2 6.u	8.0 8.1
167	, 10	0.0	0.8	0.9	5.8	5.7	ן מ	1.6	≱ Øī	6.0	8.8
148	1 >>	0.6	0.2	0.8	5.7	3.3	*	1.6	900	6.0	7.0
199 190	1 35	0.8	0.1	0.7 1.0	3.2 3.0	5.8	6) 7)	1.6	900 I	2.6 5.5	5.6 8.8
15:) <i>;</i> ;	0.8	0.1	0.7) B	2.0	Ϋ́ j	1.6	901	3.6	6.7
158	57	0.2	0.2	1.0	3	5.0	n	1.6	301	5.2	7.5
179	10	3.6	6.5	3.6	5.a	6.9	1 or	1.6	900	5.2	6.8
363 364	4 4	- }	Į	i			}	1	800 801	1.0 1.0	6.8 6.8
267	65	1				1	- 1		848	5.4	7.0
(10 mars) 361		0.1	Truss	•.s {	0.2	0.6	3 LZ	167	8:4	5.5	1.42
10 (B-8, PL/AL ₂ O ₂)	1.3.	4.1	Trece	0.5	0.5	3	313	147	MO5	vTh	P.62

a) MCII purity was 99.946.

applicate of liquid products by OLC.
 a) Some of those values are error-overly high jue to the preserve of a cultilizing activat added with the natalysis

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to improve the sclubility of organometallic compounds. Solvent agents which have been used for this purpose are listed in <u>Table 38</u>. A number of new catalysts have been prepared or purchased and are now available for testing.

Table 38. CANDIDATE SOLUBILIZING AGENTS FOR DISPERSED PHASE CATALYSTS

Isopropyl Alcohol Acetone

Methylene Chloride Dimethylformamide (DMF)

Phorone Dimethylsulfoxide (DMS)

Morpholine Piper, Jine

2-Nitropropane Butyric Acid

Toluene n-Heptane

Ethyl Acetate Hexamethylphosphoramide

1/3 sach: DMF, Acetone, n-Heptane

However, we have been involved recently in a reassessment of our experimental approach. The type of test described above, utilizing the 350 ml stainless steel autoclave, was only intended to be a way of taking a first look at catalyst activity. The present method suffers from the very slow heating rate, possibly over an hour being required to reach a 900°F steady-state test temperature. This is undesirable since the gradual heating regime of these tests may be detrimental to the reactivity of some of the catalysts, which would be true, for example, if the active form of the catalyst were in the nondecomposed state. We would prefer to heat the autoclave to its reaction temperature, and then add the reactant and catalyst.

A further complication with the original bomb test involved the accessibility of the reactant mixture and product vapors to the upper recesses of the stirring mechanism. Since the Magne-Dash required water cooling, the result has been a comparatively cold zone in the top of the bomb where condensation could occur. Deposits formed were difficult and time consuming to remove. Moreover, back-contamination from this source could have been the cause of some anomalous results encountered in the past. In order to improve this situation, we have obtained and adapted a non-stirred autoclave. It is planned to use a magnetic stirring bar, driven from the base, to stir this autoclave. The cover is equipped with 1/4-inch openings through which liquids can be charged, so that materials can be charged after the bomb is hot. Also, no dead space exists where contaminants can build up. The new equipment is ready to be pressure tested under temperature. We will also examine the possibility of using the pulse reactor (of p. 21) for testing similar types of materials as catalysts for McH dehydrogenstion.

Fessibility Culculations on Dispersed Phase Catalysis

Our confidence in the ultimate success of the dispersed catalyst approach has been reinforced by the results of some rather simple calculations of the reaction possibilities between MCH and a molecularly dispersed catalyst in a flowing system. Conditions chosen were those of interest for a regeneratively cooled system. A computer program for a plug flow tubular reactor was adapted for a variable molar flow and used to calculate MCH conversion and temperature as a function of reactor length.

For a plug flow tubular reactor with variable molar flow the reaction rate, according to collision theory, is

$$\frac{dy}{dt} = \Omega p e^{-E/RT} \frac{1 - f_C - y}{1 + 3y} \quad \text{where } \Omega = \sigma^2 \sqrt{\frac{B\pi kT}{\mu}} \frac{N_D P}{RT} f_C, \tag{5}$$

collisions/molecule-sec; the heating rate is

$$\frac{dT}{dt} = \frac{L}{D} \times \frac{HT}{P} \times \frac{FH}{C_D} = \frac{\Delta H_d}{C_D} \times \frac{dy/dt}{1 + 3y}$$
 (4)

where heat is transferred through the tupe wall at a constant heat flux, FH, and the velocity is

$$\frac{dL}{dt} = \frac{4}{\pi D^2} \times \frac{RT}{P} \times n_{T_0} (1 + 5y)$$
 (5)

The variables used in the calculations were taken from previous work and are consistent with those used in the packed bed reactor calculations:

1) Heat capacity,

for MCH
$$c_p = 79.1 + 0.0363(T - 1460 ^R)$$
, Btw/mole- R
for H₂ $c_p = 7.099 + 0.00272(T - 1460 ^R)$
for toluene $c_p = 55.9 + 0.0236(T - 1460 ^R)$
total stream $c_p = \frac{26.1 + 4.44y + (0.0363 - 0.0043y)T}{1 + 3y}$

2) Heat of dehydrogenation,

$$\Delta H_d = 92,500 \text{ Btu/mole at 1460}^{\circ}R$$

= $92,500 + 1.90(T - 1460^{\circ}R) + 0.0022(T - 1460^{\circ}R)^2$

3) Tube diameter,

D = 3/8 in. (cross-section area = 0.00110 ft²)

a) The program was developed and the calculation made by Dr. Roger Hite.

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4) Initial flow rate,

$$n_{T_0} = 928 \text{ moles/hr-ft}^2$$

= 1.08 x 10⁻⁴ moles/sec (IHSV = 103)

5) Heat flux,

$$F_H = 5 \times 10^4 \text{ Btu/hr-ft}^2$$

6) Initial temperature,

7) Tube length,

L = 10 ft

8) Activation energy,

$$E = 12.0 \text{ kcal/g-mole}$$

9) Initial pressure,

P = 1000 psis (assumed constant)

10) Collision frequency, A

Assuming molecular diameters (σ) of 6.1 A for MCH and 3.8 A for the catalyst, the collision frequency is

$$\Omega = 11.26 \times 10^9 \frac{f_C^P}{T}$$
, collisions/molecule-sec

- 11) Collision efficiency, p.
- 12) Concentration of catalyst, fc, mole percent

The results of several calculations are shown in Table 39 below. Since the reaction rate is proportional to the two unknown parameters p and for these have been lumped together in the table so that the yield, temperature, and space velocity are presented as a function of the combined parameter pfo. In Figure 20 the results have been plotted assuming a catalyst concentration of 17m, which is a feasible concentration for a cheap catalyst (such as CuO). This shows that even at very modest collision efficiencies high reactivities are indicated in the temperature region of most interest to us (500-1200°F).

Additional mathematical exploration of this system will be done in a future period.

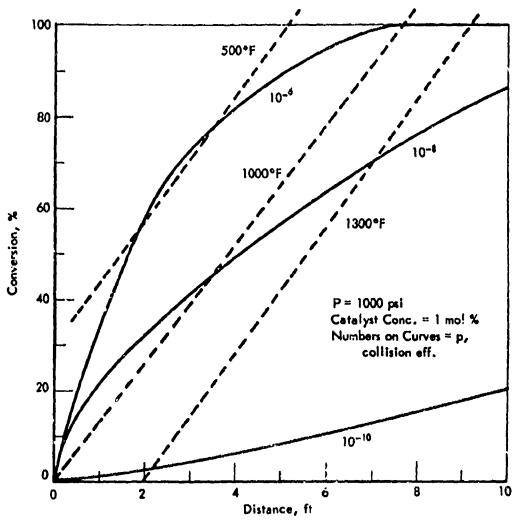


Figure 20. CALCULATED DEHYDROGENATION OF MCH BY VAPOR PHASE CATALYSIS

Table 39. MCH DEHYDROGENATION: VAPOR PHASE CATALYSIS

Distance, ft	2.5	5.0	7.5	10.0
Results for pf _C = 10 ⁻⁶ Temp, °F Conversion, °F Time, sec	491.0	635.5	946.7	1330.0
	67.4	69.9	99.9	100.0
	0.70	1.19	1.54	1.79
Results for pfc = 10 ⁻⁶ Temp, °f Conversion, 's Time, sec	947.9	1125.2	1330.9	1565.6
	38.0	57.5	73.7	86.2
	0.72	1.18	1.52	1.77
Results for pf _C = 10 ⁻¹⁰ Yemp, °F Conversion, % Time, sec	1340.2	1625.2	1882.8	2125.3
	3.3	8.8	15.1	21.4
	0.98	1.71	2.27	2.71
Results for Pfg = 0.0 (no catalyst) Temp, °F Time, sec	1371.8 1.01	1695.7 1.85	1986.5 2.57	2252.7 3.21

Thermal Stability

Tube Deposit Rating Methods

With the objective of providing a piece of equipment which would combine a standardized test of fuel thermal stability and also of catalyst activity, the Catalyst and Fuel Stability Test Rig (CAFSTR) was designed and built last year. However, the CAFSTR heat exchangers were of similar construction to the standard ASTM-CRC coker preheater, and the assessment of fuel stability depended upon visual deposit evaluations. In conventional coker tests, these ratings are made under standard lighting conditions, by comparison with standard color code penels. While this system has served acceptably for the go, no-go type quality specifications of commercial and military jet fuels, it is basically unsatisfactory for research and development purposes because of the subjective nature of the judgment which must be rendered and the different shades of color that can be assumed by similar amounts of deposits. Moreover, in non-coker tube type heat transfer studies, Burggraf and Shayesom** observed that the surface color bore no relation to loss in heat transfer coefficient.

Even within its intended scope of application, the coker's poor test repeatability has required refineries to produce fuels substantially more stable than the minimum required by the specifications; and burgeoning jet fuel demands have recently brought the need for a more quantitative fuel stability test into sharp focus.

Our own studies in the CAFSTR, where high temperatures ruled out the use of aluminum tubes, have been hampered by the development of colors of

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the tube metals themselves. Incomel 600, the alloy chosen for the construction of the CAFSTR heat exchanger tubes, changed color when heated, even in the presence of helium. Similar experience has been encountered with stainless steel. These colors are presumed due to surface oxidation of the metal at high temperatures, even when only trace amounts of oxygen are present.

In recognition of these critical tube deposit rating problems, we have been exploring several different methods of quantitative measurement, which include the following: 1) direct heat transfer coefficient; 2) combustion and absorption; 5) radiative; 4) infrared; 5) solvent dissolution-gravimetric; 6) electron micrography. This will be discussed separately.

Direct Heat Transfer Coefficient Measurements

The idea of using this as a quantitative method of deposit evaluation arose from the fact that fuel deposits can reduce overall heat transfer coefficients.

Calculations based on Zengel's⁴²) published experimental results on the effect of deposits on heat transfer, and also on the estimated precision with which the limiting variables could be either controlled or measured, led to the conclusion that deposits as thin as 0.0001 inches (0.1 mil) should be detectable. This deposit level corresponds to a coker code rating of about 2, assuming considerable coverage of the surface.

Initially, a low temperature heat exchanger method, involving a stirred calorimeter, was developed. The test utilized an actual CAFSTR heat exchanger tube and heating element, which together with a cooling coil was immersed in the calorimeter. Heat transfer measurements were made when the calorimeter liquid temperature had reached a steady state. Actually, the wall to liquid temperature differential was monitored, using a Leeds/Northrup potentiometer, capable of reading to 1 x 10⁻⁴ my (ca 0.004 °F).

Control of the voltage, cocling water, and temperature variables was recognized as of utmost importance. Voltage control to within $\pm 1/20$ volt at 110-120 volts was achieved by the use of a Superior Electric Company model IES9101 Stabiline Voltage Regulator. This instrument was used both to supply power to the CAFSTR tube and to the cooling water pump. Cooling water was supplied from a modified Colora Ultra-Thermostat constant temperature bath, and the bulk temperature was controllable to ± 0.08 °F at 90-95°F. The water pumping rate was about 2200 ml/min.

Although the bulk liquid temperatures could be held acceptably constant, difficulty was experienced with transient local temperatures in the stirred calorimeter, which eventually led to the abandonment of the calorimeter in favor of an annular heat exchanger arrangement.

In all experimental set-ups, comparisons were made of the AT (wall to liquid) for the initially clean tube and for the same tube after spray coating with (usually) 0.1 to 0.2 mil of acrylic lacquer. Our experience has been that this method, because of experimental error limits, is not sufficiently sensitive for the rating of deposits up to about the ASTM Code 4 level, but might be used for heavy deposits.

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However, it is not at all certain that failure the light deposits is entirely due to difficulty in maintaining adequately constant heat flows. There exist uncertainties as to the theoretical effects of low level deposits on the actual hoat transfer phenomena, and these effects are dependent upon the actual mechanism of deposit buildup. For example, if the deposit is not laid down as a coherent film, but as scattered particles, the increased surface roughness could cause greater turbulence near the surface. Thus, Sherriff10) has shown that as surface roughness increases, the heat transfer coefficient also increases until the heights of the roughnesses reach the thickness of the laminar sublayer. The opposing effects of low deposit thermal conductivity and increased surface turbulence can have a wide range of nat results in the overall heat transfer coefficient, thus destroying the usefulness of direct heat transfer measurements as a light-deposit rating technique. However, thick doposits, which form coherent layers, would reduce the heat transfer coefficient, and hence this technique would find application under such conditions; or, if test periods were protracted, heat transfer could be used as the basis for thermal stability rating. The significant philosophical question as to whether light or heavy deposits would better reflect the true performance of a fuel in a jet engine could be answered only via correlation studies which are beyond the scope of the wesent consideration.

Complete Combustion of Tube Deposits

A method involving complete combustion of the deposits to $\rm CO_2$ and $\rm H_2O$ has been devised and tested. On paper, combustion offers one of the best means of rating tube deposits, having the advantage of being nearly 100 times as mensitive as the direct heat transfer coefficient method. By this method it is possible to detect deposits on a 13-inch, 5/8" diameter tube as thin as 0.001 mil uniform thickness. Since a tube code rating of 0.5 is estimated to correspond to a deposit thickness of 0.025 mil, any visible deposit should be ratable by this technique.

A schematic diagram for the combustion tube rater is shown in Figure 21. Oxygen (or air), predried and purified of any indigenous CO₂, is passed through the annular combustor with the CAFSTR preheater tube comprising the heating element. Products of the deposit combustion are trapped in suitable weighing bottles, as shown, the CO having been converted to CO₂ by passage over hot cupric oxide. CO₂ and H₂O are determined gravimetrically and back calculated to determine the total deposit weight and the coefficients in the deposit formula, CxHy. Sulfur, if present is picked up in the GuO furnace as CuSO₄, while nitrogen is trapped in the Ascarite weighing tube as KNO₃. However, the pure hydrocarbons which we are testing are probably too low in sulfur or nitrogen to cause serious error from this source. In cases of high S content fuels, the S content of the deposit could be determined by passing H₂ through the CuO tube at high temperature and converting the CuSO₄ to H₂S which could be trapped in caustic and titrated or otherwise determined.

On initial startup, the "clean" apparatus required several days operation at 1000°F with gas flowing to reach a steady "zero" base line, i.e., a condition where no $\rm CO_2$ or water were being absorbed in the weighing tubes when deposits were known to be absent. This was undoubtedly due to oil adsorbed on the surface of the apparatus and to surface carbon. Cylinders of water pumped $\rm O_2$ and $\rm N_2$ are used instead of house service gas supplies to avoid introducing $\rm CO_2$, $\rm CO$, oil fog or other contaminants.

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Following the break-in period, several runs were made on artificial deposits comprising weighed samples of polystyrene. Details of these tests are tabulated in Table 40. Also tabulated are the average deposit thicknesses of these "deposits", calculated as if they were uniformly spread over the surface of a 15-in. length, 5/8-in. OD tube. Of course, in actual CAFSTR and coker tests, the deposits are far from uniform, and the maximum local deposit thicknesses may be 2 or 5 times the average thicknesses. The code ratings listed in the table were estimated based on comparative data from coker and Minex Heat Exchanger tests by Zengel. (42) While the assumed correspondence between these two tests may be poor, the heat exchanger deposits do offer an approach to estimation of deposit thickness at various code ratings. Assuming a linear correlation, the average deposit level for a 1.0 code rating would be about 0.05 mil.

Certain drawbacks exist for the combustion method, however. First, as noted, a temperature of 1000°F or more is required to burn off the deposits. Aluminum and most of its alloys are immediately eliminated, therefore. Incomel Alloy 600, (ca 72% Ni) of which the heat exchanger tubes for the CAFSIR are constructed, is somewhat oxidized at 1000°F; strongly at 1100°F. Stainless steel also oxidizes at these temperatures. While these metals can be cleaned and polished, or purhaps even reused without polishing, the possible effect of surface metallurgical changes on test repeatability would have to be explored. After several hours at high temperature, particularly at 1100°F, the Incomel tubes develop a pronounced cellular grain structure visible to the naked eye. This cellular appearance disappears when the tube is polished, however.

Table 40. COMBUSTOR TUBE RATER RESULTS ON POLYSTYRENE "DEPOSITS"

Tube		Cas	De	posit	Estimated ⁸⁾	Veight of
Wall Temp,	Flow, ml/min	Composition,	Wt, g	Average Thickness, mil	Max Coker Code Rating	Deposit Recovered, a) or Orig.
1000	350	77	0.0348	.083	1,0	104
1000	250	28	0.0144	.034	0.5	96
1000	250	28	0.0043	.010	0	107
1000	250	46	0.0105	.025	0.5	104
1100	250	46	0.0142	.034	0.5	100
1000	200	48	0.0365	.087	1.5	69

a) Calculated from CO2 + HgC recovered.

b) Estimated from data of Zengel.

建筑工工局的表面了一个

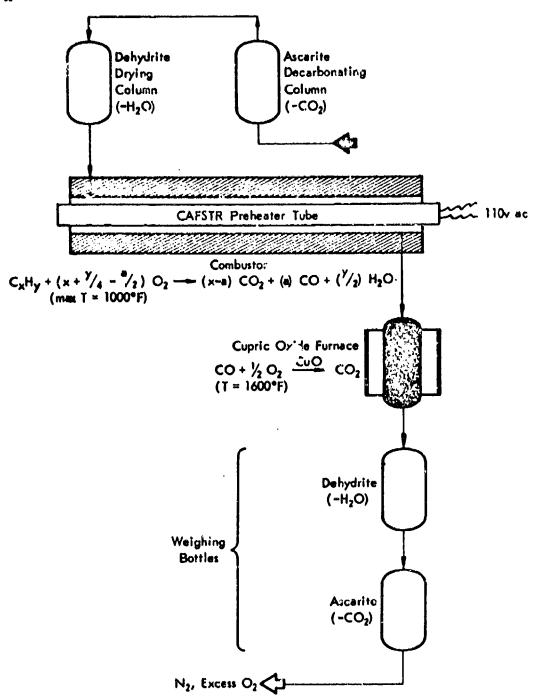


Figure 21. COMBUSTION TUBE RATOR DESIGN

A further difficulty was encountered when stainless steal preheater tubes of an integral heater/rating-tube construction from the Erdco Coker were used. The heater rapidly shorted-out internally causing the circuit fuse to blow. A second attempt with a new tube resulted in a similar experience. Apparently, these tubes were not designed to withstand the 1000-1100°F temperature level to which they were exposed. In our use of them in the Erdco they had probably never exceeded 800°F metal temperature.

Numerous runs have been made to establish optimum test conditions, the trade-off factors being time, temperature, oxygen concentration and total gas flow. A maximum rating time of 2 hours seemed to be a reasonable compromise. This allows about 1/2 hour heating-up time and 1-3/4 hours at oxidation temperature. Experimentation with temperature has shown 1000°F to be the practical minimum to obtain deposit burning. Then, for 1000°F and 1-5/4 hours, about 7% O₂ at 350 ml/min total flow is required to get complete combustion. Extremely heavy deposits would require still higher O₂ concentrations. At 40% O₂ and 200 ml/min total flow, for example, only 6% recovery was obtained (see Table 40), although this amount of oxygen is matisfactory for lower deposit levels.

A further operational problem involves removal of all traces of liquid test fluid from the preheater tube prior to rating. We had previously done this by rinsing with n-heptane and drying with nitrogen. However, the combustion tube rater reveals the presence of residual hydrocarbon when a clean tube has been rinsed and dried in this manner. Tests have shown that at low deposit levels these hydrocarbon residues can introduce a significant error. Present evolved practice is to rinse the tube three times with methylene chloride dry thoroughly with nitrogen, install the tube in the combustor and warm it to 100°F under 28 in. Hg vacuum for an hour, then start the gas flow and begin the combustion procedure.

A new variant on the burning method is now being investigated, in which the heat is supplied by an external furnace rather than using the internal heating element of the CAFSTR tube. It is hoped in this way to avoid overheating of the heating element, and possibly reduce oxidative attack of the surface metal. To this time, however, the background level of carbon, apparently arising from the furnace itself, has not been reduced to a satisfactory level.

The possibility of substituting GLC for the gravimetric procedure and using a laser as a heat source is also being considered.

Radiative Methods

Two methods of radiative deposit rating have been considered. First is that of direct beta radiation absorption by the deposit layer. This could be accomplished by impregnating the tube surface with a radioactive isotope such as Ni⁵³, but because of the possibility of catalytic effects on fuel deposition tendency, it would be preferable not to treat the tube surface itself. However, the direct radiation absorption technique does offer advantages in simplicity of application and should be explored.

A second approach to the radiative technique is that of β -radiation back scattering, using an external source. This method has the advantage of

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noncontamination of the tube surface, but the disadvantage of greater complications in implementation. Either Ni⁶³ or S³⁵ would seem to be good β -ray sources for this purpose.

Radiation scanning has the general advantage of being nondestructive to the leposit. Thus it would be possible to obtain comparable color code, combustion weight, and radiation counter ratings on the same tube. The radiation method also offers the advantage of being harmless to the tube metal and hence would not influence the life of the tube. It is believed that either method would be capable of detecting a 0.001 mil average film thickness, which is below the visible detection level but less sensitive than the combustion method. It is planned to scan the entire tube surface and to obtain an integrated radiation count reflecting the total deposits.

infrared

Some thought has also been given to the development of an infrared method of tube rating, on the assumption that it would register the effects of deposits present without reflecting influences of surface film structure. The first attempts at utilizing this idea have been discouraging, however. Using the tube itself as the source of infrared, photographs were taken of the tube in a totally darkened room. Although a fast infrared type 415 Polaroid film was used, minimum conditions for the detection of even a trace of radiation were 5 minutes exposure at 600°F tube surface temperature. This was for a stationary tube. If the tube were rotated, as planned, to photograph the entire tube surface, a much longer total exposure time or higher temperature would be required.

Even 500°F may be too high a temperature to avoid detrimental deposit changes. Other problems are numerous. The temperature is far from uniform because of end effects. Optical density of the photographic image can be affected by the eccentricity of the tube axis as it is rotated in the chuck, and the chuck drive mechanism itself must be ultra smooth since the camera shutter is open constantly during the rotation. The power cord and thermocouple wires must not interfere with the free rotation of the tube, and the power input must be precisely repeatable for both the clean tube and deposit coated tube tests. Exposure times, film properties, film developing procedures, solution strengths, and camera setups must be duplicated accurately.

Having once achieved adequate control of the variables, the precision of optical density measurements must be achieved, which of course, we have not yet been in a position to do. Some means are available for improving the photographic sensitivity of the method, such as special film processing, use of higher speed film, longer exposure times or wider slit opening, reduction of the image size by changing the focal distance, and use of ϵ faster lense; but whether these changes will accomplish the desired sensitivity is not known. The method is obviously still very much in a rudimentary stage.

Solvent Deposit Removal

Coker tube deposits cannot be recovered mechanically without the occurrence of tube damage and metal removal. It would therefore be desirable if this could be done by solvent washing. The deposits could then be recovered from the solvent and determined gravimetrically or by other means. Previously

we had tried deposit removal using readily available laboratory solvents. This was done simply by wiping with a cloth dampened with the solvent at room temperature, but all such attempts were totally unsuccessful.

Since our own and the reported experience of others indicated that coker deposits of the hard, tenacious type would not be easily removed with solvents, we decided the solvent would have to be hot if it were to work at all. The deposits could then be determined by steam jet or microgum techniques.

Our experimental apparatus consists of a coker-type annular heat exchanger in which the inner coker tube is the heat source. Approximately 50 ml of test solvent is required to fill the annular space, and after filling the device is sealed, with as little air as possible occluded. A stainless steel type pressure gage is attached, and pressure readings are taken throughout the period of the test. Arbitrarily, a temperature of 100°C was usually chosen as the maximum temperature and 500 psi as the maximum allowable pressure for the first series of runs.

Deposits were formed from either Decalin or jet fuel in an Erdoo Coker and were of a hard, nonwipeable type, usually obtained at temperatures above the break point.

The experimental approach has been to select a synthetic resin structure resembling the presumed deposit structure (as deduced from the literature)⁴⁵⁾⁴⁶) as closely as possible. Then, from published solubility parameter data on resins and solvents,⁴⁷⁾⁴⁸⁾⁴⁹⁾⁵⁰) we have selected candidate solvents.

Results to date are tabulated in Table 41. Although the objective has not been accomplished, some interesting results were observed.

Most pronounced in its action was dimethylformamide. However, dissolution was not complete and in most cases there was still a small amount of deposit left; deposits removed were not entirely dissolved but some were merely loosened. We did find, however, that a second solvent treatment on the same tube would complete the deposit removal. A few more severe (192°C) and lengthy (2 hr) treatments removed more of the deposits but still left a few dark spots.

Under the conditions selected, none of the other solvents tested were effective in deposit removal to the extent that a recognizable improvement in deposit color code rating could be observed. However, several of the solvents became darkened in color, suggesting that some deposits might have been removed. Where this discoloration was observed, the temperatures were generally not high enough to have caused solvent decomposition or exidation, unless strong catalytic effects existed.

Although M. W. Shayeson⁵¹⁾ of General Electric Company has reported that morpholine has been found to be effective in deposit removal under some conditions, we did not find it so under the conditions of our tests.

Moreover, morpholine, in our tests, appeared to have the effect of a "developer" on the deposit appearance, i.e., to cause them to look darker,

Table 41. SOLVENT DEPOSIT REPOVAL TESTS
50-54 ml solvent surrounding a coker tube

Solvent	Temp,	Press.,	Time,	Tube S	lating	Comments
	°C	palg	hr	Before	After	401924(114
Dimethyl sulfoxide	100	10	2	3.5	3.5	No effect
Phorone	100	16	1	3.5	3.5	No effect
Morpholine	100	27	2	4+	7.5	Darker
Piperidine	100	61	1	3.5	3.5	No effect
2-Nitropropane	90	200%)	1	3.5	4.5	Darker
Discetone alcohol	100	22	1	3.5	3.5	No effect
Amyl acetate	100	25	-1	3.5	3.5	No effeat
Propyl alcohol	100	102	1	3.5	3.5	No effect
sec-Butyl alcohol	100	200	1	3.5	3.5	No effect
Methylene chloride	80	198	1	3.5	3.5	No effect
Agetone	100	180	1	3.5	3.5	No effect
Methyl ethyl ketone	60	200	1	3.5	3.5	No effect
Toluene	100	190	1	3.5	3.5	No effect
Preon-II	00	200	1	3.5	3.5	No effect
Mesityl oxide	100	3	1-3/4	3.5	3.5	No effect
Tetrahydrofuran (THF)	100	170	1	3.5	3.5+	Slightly darker
Hexylene glycol discetste	60	190	1	3.5	4.8	Darker
N,N-Dimethyl formamide (DMP)	100	32	3/4	3.6	3.5	Patches remained; most areas removed
DHO!	100	26	1	3.5	0	decond consocutive treatment
D KT	100	28	1	4.0	1	Mostly removed
DHOP	100	25	2	2	0	Second consecutive
LIG 1/3 DIGF + 2/3 TiGF	142 90	180 190	2 1	4.0	3.0	treatment Much cleaner overall No effect
1/2 DKF + 1/2 morpholine Shell solutizer5) Sulfolune	100 100 100	90 25 6	1 1 1	4.0 2 2.5	7.5 3.5 2	Much darker Darker Possible slight improvement

a) Pressure increased during run suggesting occurrence of decomposition.

b) 8 m Kon, 3.1 m KIB.

without (we think) actually being of greater mass. The morpholine itself also turned dark. We have not tried this experiment without the deposit or metal environment to see if similar darkening of the solvent occurs. A mixture of half DMF and half morpholine appeared to have the same effect as morpholine itself.

Other solvents, such as 2-nitropropane, hexyleneglycol diacetate, tetrahydrofuran, and Shell K-2 Solutizer Solution (6.0 N KOH, 3.1 N K isobutyrate), also had this same apparent developing effect upon tube deposit color. The very fact that the deposits can thus be made to appear darker is further evidence of the inadequacy of the color code rating technique since different fuels might also vary in this property.

A further interest in solvent deposit removal is in the possible disclosure of chemical structural information about a deposit from solvent selectivity. Consequently, solvents selected for testing have covered a wide range of solubility parameters. (47)(48)(49)(50)

This effort is still being continued with respect to solvent selection, and will also be partially repeated with respect to higher test temperatures and longer exposure times for promising solvents.

El- in Micrography

The possible use of electron microscopy for tube deposit rating is being considered, though the current intent is to merely "take a look" to see what the observations may suggest. We are thinking here in terms of deposit structure, and perhaps there is a chance for quantitative measurements as well.

We haven't yet looked at tube deposits, but Figures 22 and 23 show scanning electron micrographs²) of stainless steel coker filters at low magnification. Figure 22 shows three shots at magnifications of 60x, 100x, and 300x of the clean filter. Figure 23 shows the same filter at 100x, three different areas, after running SHELLDYNE at 6 lb/hr and 675°F. All that can be seen by comparing these two series of photographs is how completely the filter has become plugged with debris. However, we are now preparing to repeat this comparison at about 20,000x, with the hopes of seeing the microstructure of the deposit layer on the stainless steel filter particles. We then plan to take the same sort of photographs of some sawed-up sections of a coker tube which has been previously rated on the standard Tuberator scale.

A chief disadvantage of this approach as a routine method would be its inconvenience. Also, the tube would have to be destroyed, since sections of only about 1/2 inch maximum length can be observed. The cost would be a futher deterrent. This arises not only from the cost of the electron microscope itself, but also from the laboratory preparation of the surfaces which must be made; i.e., an evaporative coating of about 75 A gold is applied at a 30° angle while the sample is being rotated. Total cost would be about 35 dollars per photograph, which must be multiplied times the number of shots necessary to describe the entire tube.

a) The electron microscopy was done by R. G. Meisenheimer.

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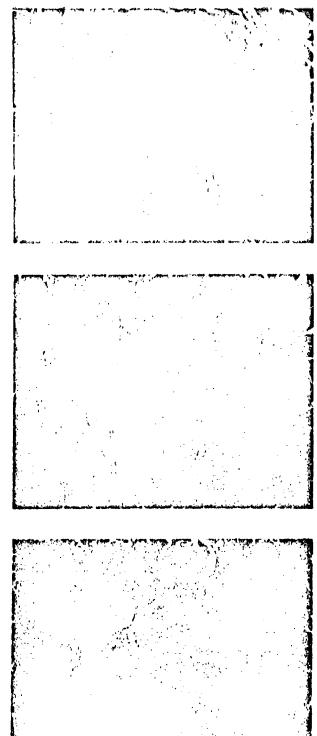
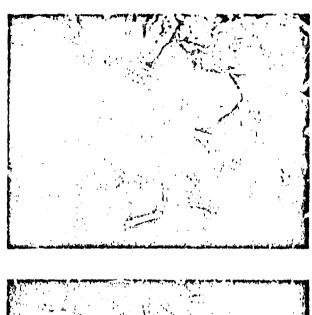


Figure 22. ELECTRON MICROGRAPHS OF A CLEAN STAINLESS STEEL COKER FILTER
Element at 60X, 100X, and 300X Magnifications



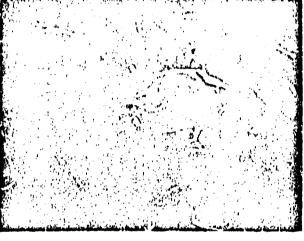




Figure 23. ELECTRON MICROGRAPHS OF A STAINLESS STEEL COKER FILTER Element at 100X Magnification, Following Run with Shelldyne at 6 lbs/hr and 675°F in the 5D/M-7 Coker

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Summary of Deposit Rating Methods

A comparison of the ASTM color code, heat transfer coefficient, combustion, radiation, and solvent deposit removal tube rating methods has been compiled in Table 42. Insufficient information is available to estimate detection levels for the infrared method.

By way of summary, it can be seen that the combustion and solvent removal techniques offer the greatest sensitivity. However, the combustion method suffers by reason of harmful tube metal effects, while the solvent method depends upon finding a suitable solvent or reactant. Beta-radiation detection ranks text in sensitivity, but the details of this approach have yet to be worked out. The measurement of heat transfer coefficients provides a sufficiently sensitive method on paper, but has not proved satisfactory experimentally. Further investigation will be necessary to select the most favorable of these methods.

Table 42. ESTIMATED MINIMUM DETECTION LEVELS FOR FIVE DIFFERENT CONCER TUBE RATING TECHNIQUES

Rating Method	Average Deposit Thickness, a) mil	Total Deposit Veight, mg	Approx ASTM Color Code Equivalent
ASTM Color Code	0.02	10	0.5
Heat Transfer Coefficient	0.05	13	0,5
Combustion	0.0004	0.15	0
Bets-Radiation	0.001	0.5	0
Solvent Deposit Removal	0.0004	0.15	o

a) The maximum deposit thickness is probably 5-5 times the average thickness.

b) Based on average thickness of deposit.

Overall the combustion and radiative methods appear most promising, and at the present time most of our effort is directed toward the combustion method. We will also in the near future evaluate a method utilizing an H2 ring burner or a laser as the source of combustion, with the expectation that this will be more rapid and less destructive to the tube, and particularly, to the tube heater. The possibilities of utilizing other methods of measurement, involving IR, x-ray, and dielectric properties are being studied. The success of the CAFSTR and of future coker-type testing depends upon the solution to this problem.

Modification of the SD Coker; SD/M-7

The SD Coker, as originally designed, 1)52) operated on a fuel recycle scheme and utilized a glass double reservoir to measure the fuel flow during recycle operation. This device was chosen to avoid the calibrations required with rotameters when using experimental fuels with a wide range of densities. A description of the operation of this double reservoir device has been given previously. 52)

Although the current system has been simple to operate, and satisfactorily accurate, there have been two notable defects. First, the glass reservoir and joints were subject to occasional breakage; and there was always the safety hazard that failure of the Grove pressure regulator might release full system pressure into the reservoirs and explode them.

A second problem had more to do with the inadequacy of the Zenith pump used than with the glass reservoir system itself. Particularly with lower viscosity fiels such as methylcyclohexane, pump wear has been both severe and variable. Generally, the higher the pressure developed the greater the pump wear, but this differed with individual pumps.

However, since the mating perts are machined to a tolerance of about 0.0002 in, the Zenith pump is a satisfactory metering device. Only when producing high pressures with low viscosity fluids does scuffing difficulty develop.

In our CAFSTR^{a)} and in the modified Erdco Coker we have gone to a gas pressure drive, which is a good system, but not readily applicable to recycle operation (which we favor because of the often limited availability of experimental fuels).

We have now modified the SD coker to eliminate the glass reservoirs. The revised system, shown in Figure 2h is designated M-7.

In this arrangement the d/p cell, besides reading pressure drop across the filter, serves as a fuel reservoir and sparge for equilibration with the pressurizing gas. The pump serves only to meter the fuel and recirculate it. Since the pump is of a volumetric displacement type, the flow rate is determined with an electronic counter which reads in tenths of rym the rotation of the pump drive gear. Total system pressure is supplied by the sparge gas, sparging action being maintained by a slight imbalance in the settings of the sparge gas supply and system pressure regulator settings. The sight glass permits instant visual observation of the sparging rate and also doubles as a surge. The sparging rate can be observed at the Grove regulator gas discharge tube, where a simple bubble flow meter is used to quantitatively measure the sparge gas rate. A standard rate of loo al/min has been found satisfactory.

Since the entire fuel system is under pressure, it is necessary to replenish the exygen reacted in the hot test zones by using a sparge gas of the proper O_2 partial pressure. Because of the effect of pressure on O_2 solubility, the required percentage of O_2 in the sparge gas is less than for

a) Catalyst and Fuel System Test Rig.

atmospheric equilibration, and varies with the pressure. For example, to provide fuel which would be oxygen saturated at 1 atmosphere requires equilibration with sparge gas containing about 1.87 \sharp C₂ at 150 rsig.

Similarly, if a low dissolved oxygen concentration is desired, the magnitude of the total pressure imposes limitations on the minimum levels attainable. In Table 45 the equilibrium dissolved 02 in shown versus pressure for 1 ppn oxygen concentration in the sparge gas. These values were calculated from Ostwald Coefficients for a patroleum fraction of 0.78 specific gravity at 60°F⁵⁰) and demonstrate the lower 02 limits which may be expected. The restrictions will not be serious, however, since even 30 ppb oxygen is difficult to measure experimentally.

Table 43. FFFECT OF TOTAL FRESSURE ON DISSOLVED OXYGEN CONCERRATION IN A 0.78 SPECIFIC CRAVITY HYDROCARBON WITH AN EQUILIBRATING GAS CONTAINING 1 PPM

Total Pressure. pei	Dissolved Op, Parts Per Billion
150	5
250	8
400	12
600	18
1000	30

Although we intend to eventually operate the SD coker at 1000 psi pressure, operation to date has not exceeded 600 psi. Suprisingly, even at 400 psi pressure a small amount of leakage occurs between the plates of the Zenith pump, though these pumps are specified by the manufacturer for use at pressures above 1000 psi (but with viscous fluids). Apparently, the phenomenon is related to the high pressure on both inlet and outlet sides of the pump, since leakage was never a problem when the pump itself was developing the system pressure and the inlet side was near atmospheric. Thus leakage could give trouble at higher pressures. It may be possible to control this by using additional bolts or clamps.

Despite these problems, the new recycle arrangement has been found to operate quite satisfactorily. The pump operates almost without noise which indicates little wear is occurring. The fuel system requires 350 ml, and is filled to a standard mark on the Jergusen sight glass at the start of each test. This level has not been observed to change during runs. Flow rate checks are rapidly and simply made during the test using the electronic caunter and have been found constant to 40.5 percent.

Because of the avoidance of wear debris from the pump, the M-7 coker may have become a somewhat less severe test than it was previously.

Effect of Metal Environment on Decalin Thermal Stability

The presence of trace metals has long been recognized as a deterrent to good thermal stability of liquid hydrocarbon fuels. Such trace metals are always present, deriving from the construction materials of fuel system components. We have found, for example, that variations in wear rates of the SD Coker Zenith pump can cause differences in tube deposit ratings, though this could be due in part to the presence of metal in the deposit. (55)

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Since trace metals are known to differ in their influence on fuel stability, 30-40) it was decided to investigate the effects on the thermal stability of Decalin of Some of the more commonly encountered metals, using the SD/M-7 Fuel Coker.

The Decalin used in these tests was prepurified by percolation through a silica gel column. Prior to the test, the Decalin was put on a laboratory shaker for a minimum of 7 hours with the chosen metal in a powdered state (ca 5 grams/liter). The powdered metal was then filtered out, using a 0.45 micron Millipore Filter, and the Decalin introduced directly into the coker. Although some tests were tried with only this pre-equilibration, the general procedure was to attempt constant re-equilibration by recirculation of the fuel through a coarser bed of the test metal throughout the test. A subsequent test was then conducted under identical conditions with about 240 ppm of the metal deactivator (MDA), N,N'-disalicylidene-1,2-propanediamine, added after filtering.

An unfortunate circumstance in the present tosts is that the coker fuel system, except for the aluminum preheater tube, is 316 stainless steel throughout, thus providing a constant background of contamination with those elements present in the stainless steel. From the typical analysis of 316 stainless shown in Table 45, the large amounts of Cr. Ni, and Fe are particularly to be noted, since these are among the metals under present investigation.

The complete list of metal environments investigated includes Fe, Cu, Ni, Cr, Zn, Pb, and additional stainless steel. All runs were at 150 psig and most were at 600°F. Preliminary results are tabulated in Table 44.

Most deleterious to Decalin thermal stability ratings were copper and iron. At 600°F both caused increases of 3 code numbers in tube ratings. One difference noted between the two metals was that copper caused a significant increase in filter plugging (11.5 "Hg compared to 2.1" without copper). Iron caused no filter plugging at all. The introduction of MDA with each metal resotred the tube ratings to the "neat" Decalin stability level (Cu/MDA, 3.5/17; Fe/MDA, 3/11; metal free, 3/22). MDA also eliminated the filter plugging observed with copper (Cu, 11.5"; Cu/MDA, 1.5" Hg). At 600°F, then, MDA nullifies all of the deleterious effects of dissolved copper or iron. When iron was run by pre-equilibration alone (no re-equilibration bed in the flow stream), the ratings were essentially the same as for Decalin alone (Fe, 3/16, 0", Fe-free, 3/22, 2.6" Hg). Cu was the only metal having a significant influence on filter pressure drop.

Chromium also had a small adverse effect on Decalin ratings (Cr, 4/33, 0.8" Hg; Cr-free, 3/23, 2.1" Hg), which was not significantly improved by the addition of MDA.

The presence of nickel had no appreciable effect, although the improvement due to MDA addition which was observed without nickel was not found with nickel (Ni-free/MDA, 2/12.5; Ni/MDA, 3/14.5;). Since the coker flow system is constructed of stainless steel, as previously mentioned, we suspect the true harm contributed by Cr and Ni may be greater than that shown by these tests.

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Table 44. EFFECTS OF METAL ENVIRONMENTS ON THERMAL STABILITY OF DECALIN (F-139) WITH AND WITHOUT METAL DEACTIVATOR (MDA)

Coker Runs	Temp,	Metal Pre- equil.	Metal Re- equil.	MDA Added, g/liter	Preheater,	Tube Max Tota	κ/ ΔP,
285, 286, 314 315, 316, 346	600	•	•	•	508	3 /2	2 2.1
255, 262 263 a , 263B	625	•	-	•	620	3 /2	23 0.0
284	650	•	•	•	686	3.5/2	£6 2. 9
320	675	•	•	•	674	5 /2	28 0 .2 ·
287	600	•	•	0.0046	500	3 /3	0.0
295	600	-	•	0.214	505	2 /	2.5 0.8
294	625	-	•	0.214	632	4.5/2	20.5 0.7
293	675	•	•	0.214	728	8 /2	20.5 0.4
288	600	Fe	•	•	499	3 /1	6 0.0
303	600	Fe	Fe	•	499	6 /4	0.0
304	600	Fe	Fo	0.214	499	3 /1	0.0
290	600	Cu	Cu	•	503	6 /4	1 11.5
291	600	Cu	Cu	0.214	505	3.5/1	17 1.5
327, 334	600	Ni	N1	•	499	3 /1	
317, 333	600	Ni	Ni	0.214	490	3 /1	¥.5 0.0
308	600	Cr	Cr	-	505	4 /3	0.8
310	600	Cr	Cr	0.214	505	3.5/2	4.5 4.8
332	600	31638	31688	•	474	2 /1	4 0.0
351, 321	600	31688	316SS	0.214	474	5 /1	o 2
319, 318, 292	600	Zn.	Zn	0.214	505	1.5/1	0 1.5
330	600	Zn	2m	•	464	-	2.5 0.2
515, 325	625	Zn	Zn	_	610	3 /2	3 1.5
296,501,302,	,					•	
511,529	625	Zn	Zn	0.21لب	634	3 /1	8 0.6
297	625	Zn	-	0.214	622	-	7.5 0.3
337	600	Pb	Pb	0.214	504	2 /1	7 0.0
338	600	Pb	Pb	-	498	2 /1	7 0.0

Table 45. CHEMICAL ANALYSIS OF 316 STAINLESS STEEL

Element	
C	0.10
Mn	2.0
Cr .	16-18
N1	10-14
Si	1.0
Мо	3.0
Fe	62-28

In view of the results for Ni and Cr, however, the data with added 316 stainless were surprising, since the tube ratings were improved (no added stainless, 3/25; stainless added, 2/14). When MDA was added, no further effect was found.

Interestingly, at 600°F the presence of Zn and Pb gave similar improvements in tube ratings to that of 316 SS, which again were not significantly altered by the further addition of MNA. But at 625°F, no includes of Zn occurred.

Further data will be needed to confirm the tentative conclusions stated here for Ni and Cr. However, the harmful effects of Fe and Cu, and the beneficial effects of Zn, Pb, and 316 SS represent deviations which are outside the 95 percent confidence limits for the determination of Decalin ratings, and are therefore considered reliable. The latter three metals appear to be removing something from Decalin which is harmful to its thermal stability, while Fe and Cu appear to behave as oxidation catalysts. The beneficial effect of MDA in the untreated and in the Cu and Fe treated cases is also cutside the 95 percent confidence limits of the pure Decalin rating, and is therefore considered real.

When beneficial effects of MDA do occur, such as with copper and iron, the improvement is probably due to its chelating action, but this must largely occur during the first few recycles of the SD/M-7 Coker, since MDA itself is not stable above about 540°F. A) However, the chelates of MDA would be expected to be stable at higher temperatures, and might therefore be expected to survive the 600°F coker exposure temperature. This may explain why MDA was beneficial without metal additive at 600°F, while at higher temperatures (i.e., 625-675°) the instability of the shelate itself might contribute to deposit formation. The data of Table 45 are in harmony with this hypothesis.

3.51/2**5.17.2.131.2.722316788**

a) MDA decomposes to phenol and other lower boiling products, du Pont MDA Bulletin. Its stability would expected to be somewhat greater in dilute solution.

Table 46. EFFECT OF METAL DEACTIVATOR (240 Pim) C.I TH TMAL STADILITY OF DECALIN

	SD/M-7 Tube Ro	
Temp,	Without MDA	With MDA
600	3 /22	2 /12.5
625	3 /23	4.5/20.5
650	3.5/26	-
675	5 /28	8 /37

SD/M-7 Coker Cleamip Procedure

Because the M-7 Coker operates on a recycle principle, special precautions have to be taken to avoid inter-test contamination. Deposits may be laid down in the cooler and in other cold regions of the flow system, which are not readily removable by mechanical techniques such as are used in the cleanup of the preheater and test filter. Moreover, low temperature deposits laid down in one run may be partially dissolved by the test fuel of the next run, thus resulting in an experimental rating error and failure to reproduce results.

To avoid this hazard, a new procedure for cleaning the system has been developed, based upon tests of the effectiveness of various solvents in removing deposits. Solvent effectiveness has been judged from both a general knowledge of component solvency power and by the color of the solvent upon its removal from the system.

Influence of Solvent Contamination on SD/M-7 Coker Ratings

Despite the extensive care taken in trying to remove all solvent from the system following the solvent wash series, it is possible that solvent contamination might result in misleading results. The use of dimethyl-formamide (DMF) was of particular concern because of its nitrogenous character, although it is regarded as a very chemically stable compound.

To determine the effects of such contamination, DMF and toluene were run in the coker at 1 percent (v) concentration with Decalin. At 600°F the ratings were DMF, 2.5/10 and toluene, 2.5/14. No filter plugging occurred in either case. This compares with average ratings of 5/23 and 2.6" Hg for identical runs without DMF or toluene. In other words, neither solvent causes harm, and may even improve the thermal stability rating of Decalin (perhaps due to doposit removal from the preheater and filter due to improved solvency).

We had also been concerned whether Decalin runs following SHELIDYNE tests could have possibly suffered from contamination with SHELIDYNE. Consequently, a similar test to that with DMF was made with SHELIDYNE as the "contaminant." Results with 1 percent SHELIDYNE at 600°F were 2.5/19.

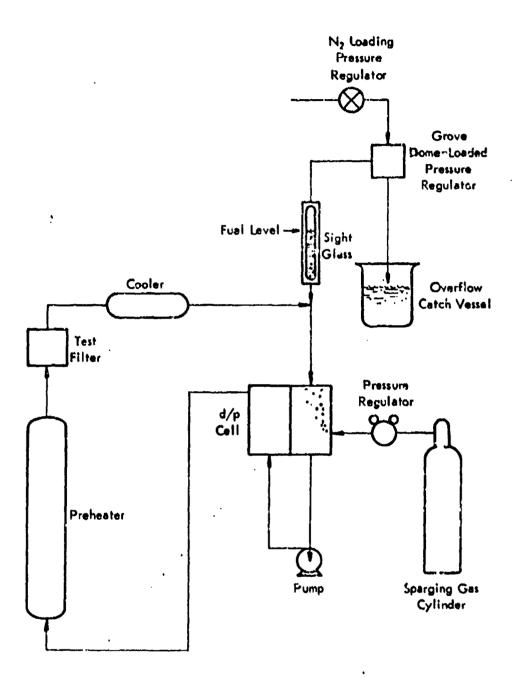


Figure 24. REVISED FUEL SYSTEM FOR THE SD/M-7 COKER

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and no rise in filter pressure drop--an almost identical rating to that with DMF.

Additional tests of this type will be made with other solvents which have been used, i.e., ethyl acetate and acetone.

Thermal Stability of SHELLDYNE and SHELLDYNE H

The thermal stability of SHELLOYNE, SHELLOYNE H, and Decalin was observed in the SD/M-7 Coker using the recycle mode. The results are given in Table 47. It will be noted that both SHELLOYNE and SHELLOYNE H have less tendency to form tube deposits than Decalin but much greater filter plugging tendencies. This latter attribute is probably exaggerated by the recycle mode used in the test, since on the average the charge of fuel is recycled 35 times through the heated zones. Even so SHELLOYNE H is substantially less prome to form both tube and filter deposits than the parent material.

A more recent sample of SHELLDYNE, although it had an adsorbence at 357 microns wave length 27 percent greater than the previous sample, also gave a satisfactory SD/M-7 Coker rating (T_{2.5} breakpoint of 600°F). Thus, the thermal stability of the SHELLDYNE samples received have been reasonably reproducible from batch to batch, despite differences in color appearance. The latter SHELLDYNL has now been hydrogen treated for use in the mini-FSSTR studies.

Thermal Stability of P and W 535 Jet Fuel

A cons-drum sample of P and W 535, a high quality JP-7 type turbine fuel, was obtained from Pratt and Whitney West Palm Beach Florida facility for use in comparative tests. This fuel is being used by P and W for most of the heat transfer testing being done at their Florida location. We have now determined the thermal stability of this fuel using the SD/M-7 Fuel Test Rig. The breakpoint temperature, $T_{2.5}$, of 600°F was found to be identical with that of Decalin. Filter plugging was almost nil (0.15" Hg). The color change in the fuel was slight, becoming a very light yellow, and low solvent coloration during the post-run cleanup indicated a low level of cold zone deposit formation.

Table 47. COMPARISON OF THERMAL STABILITY OF "SHELLDYNE," "SHELLDYNE"-H AND DECALIN

5 hr SD/M-7 Coker ratings, 6 lb/hr fuel flow rate. Recycle mode, 0g content equivalent to equilibration with 1 atmosphere air, 150 psis.

	Fluid Temp,	Filter AP, in. Hgf)	Tube Ratinga)
SHELLDYNE H	675	13.0	1.5/8
11	700	6.8	3 /22.5
#	725	-4.4	4 /23
п	(690) _b)	(10.0)b)	(2.5/20)b)
SHELLDYNE	575	53	0.5/3
n	625	ca 179	3.5/24.5
n	650	ca 305	6 /36
#	675	ca 204	1.5/10 (47 min) ^{c)}
**	(610)b)	(141) ^{b)}	(2.5/18)b)
Decalin			
F-113d)	600	0	2.5/16.5
F-139 ^{e)}	600	0	3.0/20.5
	625	0	3 /23
	650	2.9	3.5/26
	600	0	3.0/16
	600	0	3.0/15
	(600) _{p)}	(o)p)	(3.0/15)b)

a) ASTM Color Code Ratings, Max/Total.

b) Interpolated tube rating breakpoint, assumed to be at code 2.5, and corresponding pressure drop.

c) Test terminated because of rapid increase in filter pressure drop.

d) Analysis: 21.5 trans, 74.5 cis, 0.4 tetralin. e) Analysis: 33.5 trans, 65.5 cis, 0.8 tetralin.

f) Filter temperature = fluid temperature.

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Fuel System Simulation Test Rig

The Fuel System Simulation Test Rig (FSSTR) has been described in detail in the three annual reports associated with the preceding contract on this subject, 1/2/3 therefore no description of the unit will be included here. However, a flew scheme is repeated as Figure 25 for convenience.

During the past year the following studies have been conducted in the FSSTR:

- (1) Catalytic dehydrogenation of MCH over UOP-R8 in a 0.277" ID x 2-ft long reactor section.
- (2) Heat transfer to MCH in the empty 2-ft reactor section.
- (3) Heat transfer to MCH, water and Ng in 0.0265" ID x 4" and 6" long heat exchange sections.

(The last two items are part of a continuing cooling study program.)

Catalytic Dehydrogenation of MCH Over UOP-R8

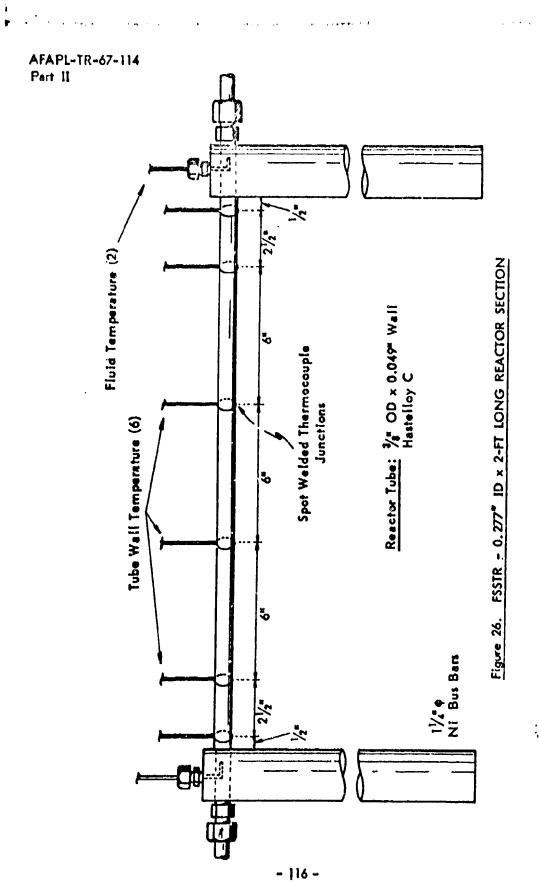
A description of the reactor used in these tests as well as a report of the initial test series was included in the immediately preceding Annual Report on this project. 19) For convenience, the description is repeated here and the results of the first test are incorporated with the remainder of the data.

0.277" ID x 2-ft Long Reactor

In order to permit investigation of heat flux conditions closer to those which might be encountered in combustion chamber cooling, a short reactor section was constructed and installed in the FSSTR in place of the usual 10-ft reactor section III. This reactor, a sketch of which is shown in Figure 26, was made up of a 2-ft section of 3/8 in. OD x 0.049 in. wall Hastelloy C tube welded to Ni bus bars. Compression type fittings (3/8 in.) provide inlet and outlet connections, and 1/16 in. fittings are used as glands for inlet and outlet fluid temperature thermocouples. A 1/16 in. fitting was also provided in the exit end bus bar for sample withdrawal. Tube wall (external) temperatures were measured at six locations by thermocouples spot welded to the reactor tube. The two lead wires from each junction (insulated from direct contact with the tube by ceramic cement) were wrapped 1/2 turn each in opposite directions around the tube and then passed through ceramic insulating tubes to points far from the high temperature area.

The 10-ft reactor sections normally used in the FSSTR use 1/16 in. OD, insulated junction, sheathed thermocouples welded to the tube wall for monitoring wall temperatures. The 2-ft reactor section made for this study was originally constructed in the same manner. During preliminary heating of the reactor with N_2 flow through the tube it became evident that, without a compensating heat tube surrounding the reactor, as is used around the 10-ft sections, heat leaks along the thermocouple sheaths were causing errors in temperature measurement. The use of unsheather 30-gage (0.010 in.) thermo-

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couples spot welded directly to the reactor was tried and proved satisfactory. A comparison of recorded temperatures obtained at the same time from the two types of couples is shown in Figure 27. The bare wire couples were used for all MCH test runs on the 2-ft reactor. Using this type of couple results in electrically hot lead wires and appropriate safety precautions had to be taken.

The electrical resistance of this reactor section is about 0.025 ohms. With the power supply presently in use, a maximum heat flux of ca 590,000 Btu/(hr · ft²) can be reached without exceeding the 1000 ampererating of the transformer secondary.

A measurement of heat losses from the reactor has been mad by passing heated N_2 through the tube and recording the fluid temperature entering and leaving and the tube wall temperature profile. With the assumption that the tube wall temperature profile is parallel to the average N_2 temperature profile, an extrapolation of a straight line through the wall temperature points to the 0 in. and 24 in. limits (disregarding the points 1/2 in. from either end which were apparently influenced by heat leak to the bus bars) yields the N_2 temperature drop along the reactor tube proper.

Subtracting this loss from the total loss (determined from overall fluid temperature drop corrected for any power input) gives the heat loss to the two bus tars. Equal loss to each bus bar has been assumed. The heat losses so determined are shown as the solid points in Figure 28. Note that the "tube" losses agree with those determined later with the tube empty but that the "end" losses are higher than the empty tube values. This is because the outside tube wall temperature determines the heat loss from the tube and it is the outside wall temperatures which are measured, while in the case of the bus bar losses, the temperature used is that of the fluid passing through the opening in the bus bar and heat flux to the bus, depends not only on this temperature but also on the heat transfer coefficient between fluid and bus which is influenced by flow rate and also by that portion of the catalyst bed which enters the bus bar. The heat flux values given in the data summary tables have been corrected for the "tube" loss. Any overall heat balance made should incorporate both "end" and "tube" losses.

Test Runs

The initial system tested using the 2-ft reactor section was MCH over UOP-R8,Pt on Al₂O₃,catalyst. Table 48 summarizes the data obtained during the six test series making up the study. Each of the series will be described briefly in the following discussions.

The first test series (10018-50) was designed to duplicate the reactor inlet conditions and power input specified as Condition B in Table 70 of the last annual report. A direct comparison of experimental data and performance predicted by the mathematical model could then be made. (This is discussed later.) A feed rate corresponding to a mass velocity of 150,000 lb/(hr·ft²), an inlet pressure of 900 psig, and an inlet temperature of ca 900°F were established and power to the reactor was increased in steps until the specified heat flux [ca 350,000 Btu/(hr·ft)] was reached. As it became evident that the catalyst was deactivating, the test was discontinued at that point.

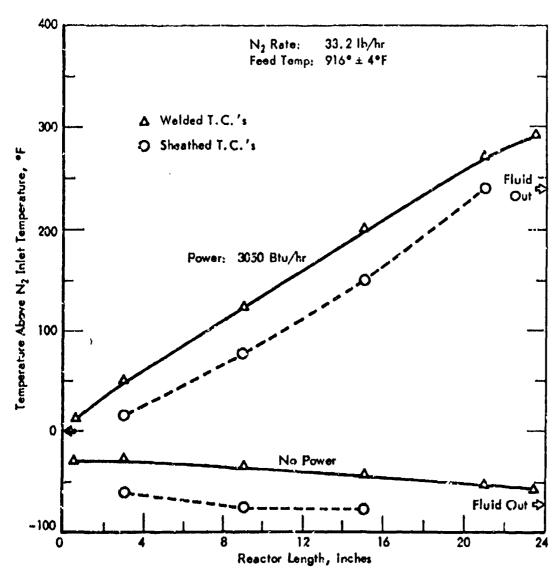
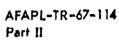
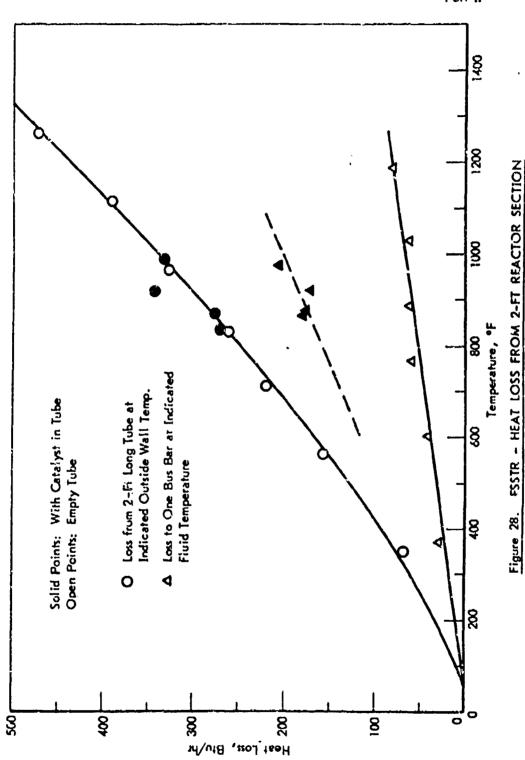


Figure 27. FSSTR - COMPARISON OF SHEATHED AND BARE WIRE THERMOCOUPLES ON 2-FT REACTOR SECTION





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FSSTR - MCH OVER UOP-RB IN 2. FT REACTOR: DATA SUMMARY Table 48.

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USV bases on superficial values of 0.277-in. ID x 2-ft long reactor. **=** =

Meas velocity based on inside cross-section of reactor.

Corrected for loss along tube. Heat flux besed on inside surface area.

Outside wall tesperature at indicated distance from start of tube.

Moles of UCH converted per 100 moles of feed. ರಕರಿಕೆ

Tube wall temp rising rapidly. See separate plot of temperature-time data.

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Table 48 (Conte-1). FSSTR: MCH OVER UNP-RB IN 2-FI REACTOR

Table 48 (Contd-2). FSSTR: MCH OVER UOP-RS IN 2-FT REACTOR

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The effect of catalyst deactivation is shown in Figure 29 where the exit fluid temperature is indicated. The continuing temperature rise shown in the final runs after a step increase in power indicates that the heat sink resulting from reaction is decreasing and a corresponding amount of power is heating the product.

Even though the catalyst had evidently been slowly deactivating prior to the final power increase, the initial conversion at that level reached about 66%. Comparing this with the predicted conversion of 57% indicated that some modification of the model would be required.

Series 10018-52 was run at the same reaction inlet conditions as the preceding series as a check on the previous results. Longer lined-out periods of operation confirmed that deactivation was proceeding at temperatures at least as low as 900°F.

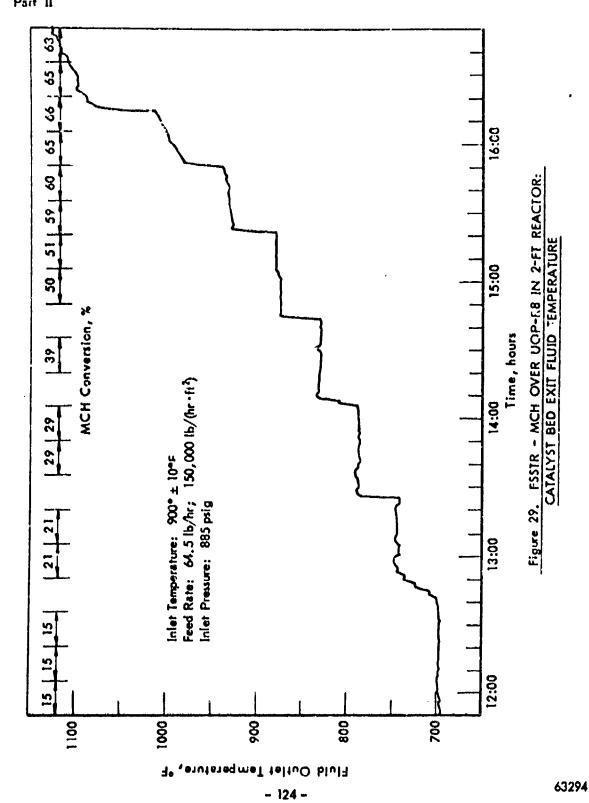
As tests in the bench-scale reactor (Figure 6)¹⁹) had shown some temporary improvement in catalyst activity resulting from hydrogen treatment, this procedure was tried on the partially deactivated catalyst. H₂ was passed through the reactor while maintaining inlet and outlet fluid temperatures at ca 1000°F for 20 minutes. No significant increase in activity could be demonstrated during the initial run conditions established for Series 10018-55. If any enhancement of catalyst activity occurred, it was very temporary and had dissipated during the time required to bring the unit to an operating condition corresponding to the final run of the preceding series. The test was continued, increasing power input in steps, until a heat flux of 376,000 Btu/(hr.ft²) was attained. At this point the tube wall temperature at a point 1/2 in. from the exit end started increasing rapidly and the run was discontinued. This will be further discussed in a subsequent section. The increasing rate of catalyst deactivation at higher temperatures was clearly demonstrated during this series. Figure 30 shows the MCH conversion-time data taken at three temperature levels.

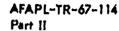
A fresh charge of UOP-R8 was then installed in the tube and activated in N_2 for 2 hours at 1100°F.

Series 10018-60 was a brief test at moderate power input made to confirm that catalyst activity was the same as for the first charge.

In order to provide further data for modification of the mathematical model the next tests (Series 10018-62) were made at a lower flow rate, $C = 60,000 \text{ lb/(hr·ft^2)}$. This corresponds to the mass flow rate used for a number of tests previously made in the 10-ft reactor sections. During this series a maximum heat flux of 168,000 Btu/(hr·ft²) to the catalyst section was reached, resulting in an initial MCH conversion of C_0 .

The final test series (Series 10018-64) was designed to reach the maximum heat flux permitted by the 1000 ampere power supply limitation, ca 590,000 Btu/(hr·ft²). Feed at ambient temperature was used for these runs and power was increased in steps as usual, until at a heat flux of 570,000 Btu/(hr·ft²) a rapid rise in tube wall temperature forced shut-down. Conversion was 47% at this condition.





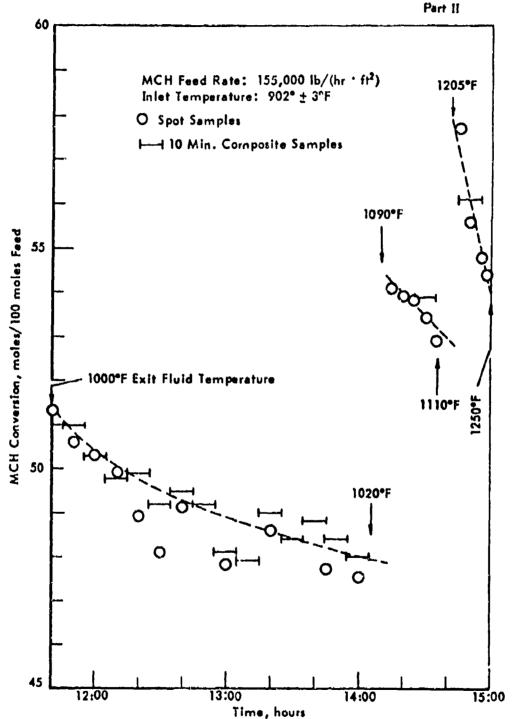


Figure 30. FSSTR - MCH OVER UOP-R8 IN 2-Ft REACTOR: DECLINE IN CATALYST ACTIVITY DURING SERIES 10018-55

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Coke Deposit: Temperature Limit

Series 55 and 64 were each terminated when a runaway temperature vieveloped after a step increase in power. The sudden increase in temperature was measured by the tube wall thermocouple located 1/2 in. before the exit end of the reactor. Figures 31 and 32 show the time-temperature data recorded during the final portions of the two series. Tube wall temperatures at 21 in. and 25-1/2 in. points and exit fluid temperatures are shown. A rapid temperature rise of this nature occurred during previous experiments on the 10-ft reactor (Figures 58 and 59),2) at which time catalyst deactivation was caused by increasing the temperature of MCH feed to the catalyst section to 1100-1150°F. However, sudden catalyst deactivation was obviously not the mechanism involved in the present problem, since the exit fluid temperature did not follow the tube wall temperature, which would have been the case if the heat sink capacity were lost due to a sudden decline in conversion. It appeared more likely that the increase in temperature difference between tube and fluid was the result of an insulating coke layer being deposited on the tube wall and a consequent decline in overall heat transfer coefficient. This was substantiated when the catalyst charge was dumped following Series 55. The bulk of the charge poured easily from the tube but it was necessary to rod out the final 1/2 in. to 1 in. portion of the bed. Examination of the catalyst particles from this final section showed a coke sheet adhering to one tangent of some of the beads. A photomicrograph (Figure 33) of a recovered particle shows the coke sheet clearly. The broken catalyst bead was probably fractured during removal from the reactor tube. The coke film in the picture is about 0.01 in. thick. No generalized conclusions can be drawn from this single case. However, for the system involved, rapid coke build-up should be anticipated in any region where tube wall temperatures of 1350°F and fluid temperatures of 1150°F might be encountered. Whether the presence of the catalyst affects these temperatures will be checked in subsequent runs.

The R8 catalyst in the runs in the 2-ft reactor has shown much more tendency to deactivate than it did in the runs in the 10-ft reactor, due probably to the higher heat flux. Work under our catalyst development program has provided catalysts of greater stability (as indicated by bench scale tests) than the R8. Enough of one of these (10280-113) has been made up to carry out tests in the 2-ft reactor. This will be done in the subsequent period.

Cooling Program, Experimental Study

A program has been initiated to provide experimental data which will yield information on the characteristic behavior of hydrocarbon fuels under conditions of very high heat flux.

Proposed targets for this investigation are:

- 1. Heat sink in the region of 600 Btu/lb (from latent and sensible heat only).
- 2. Tute wall temperatures of 1300-1400°F.
- 3. Heat flux in the region of 7-8 x 10^6 Btu/(hr·ft²).

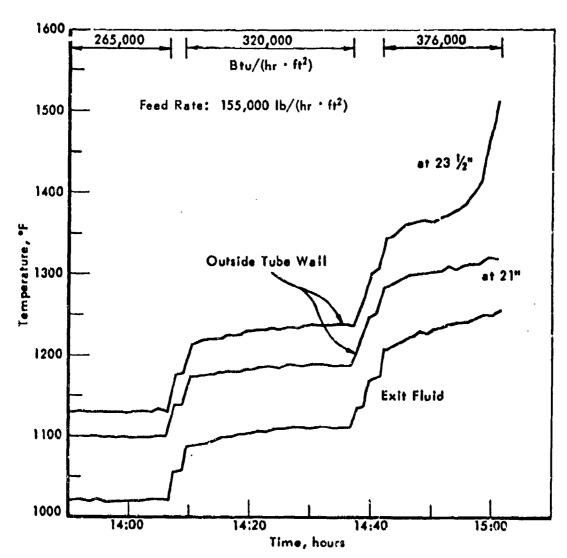


Figure 31. FSSTR - MCH OVER UGP-R8 IN 2-Ft REACTOR: EFFECT OF COKE DEPOSITION ON TUBE WALL TEMPERATURE, SERIES 10018-55

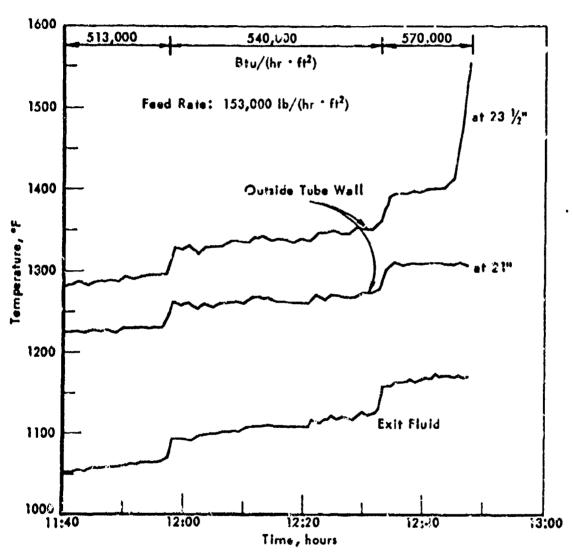


Figure 32. FSSTR - MCH OVER UOP-R8 IN 2-Ft REACTOR: EFFECT OF COKE DEPOSITION ON TUBE WALL TEMPERATURE SERIES 10018-64

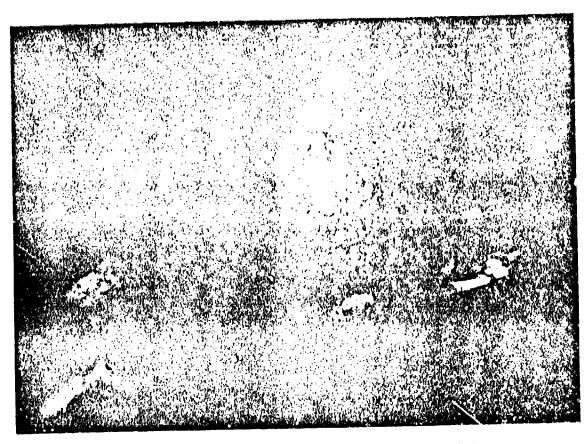


Figure 33. FSSTR - MCH OVER UOP-R8 IN 2-FT REACTOR:

COKE FORMATION FROM SERIES 10018-55

Magnification: 56X

4. Tube ID between 1/4 in. and 0.02 in.

The major objective of the study is the experimental verification of, or if necessary, modification of, heat transfer correlations for use under these extreme conditions. Also, as some thermal reaction is spt to be found at the high wall temperature levels, the possibility of coke deposition on the tube walls exists and the effect of this deposit on heat transfer as well as the possibility of plugging in the small diameter tubes will have to be investigated.

0.277" ID x 2-ft Long Heat Transfer Section

The initial effort in this study, using MCH as the test fluid, was conducted using the 3/8 in. OD x 2-ft long heat transfer section which was described previously. Some changes made in thermocouple placement will be discussed later.

Test Runs

Three series of test runs have been completed at conditions which met as many of the proposed turgets as possible while using this section. The range of variables covered was:

Tube Size; 0.277 in. ID x 2 ft long Pressure: 600-900 psig

Pressure: 600-900 psig Feed Rate: 241,000 1b/(hr·ft²) ≈ 2500 LSHV

Max Heat Flux: 163,000 Btu/(hr.ft2)

Max Bulk Fluid Temp: 913°F

Max Tube Wall Temp: 1350°F (outside) 1320°F (inside)

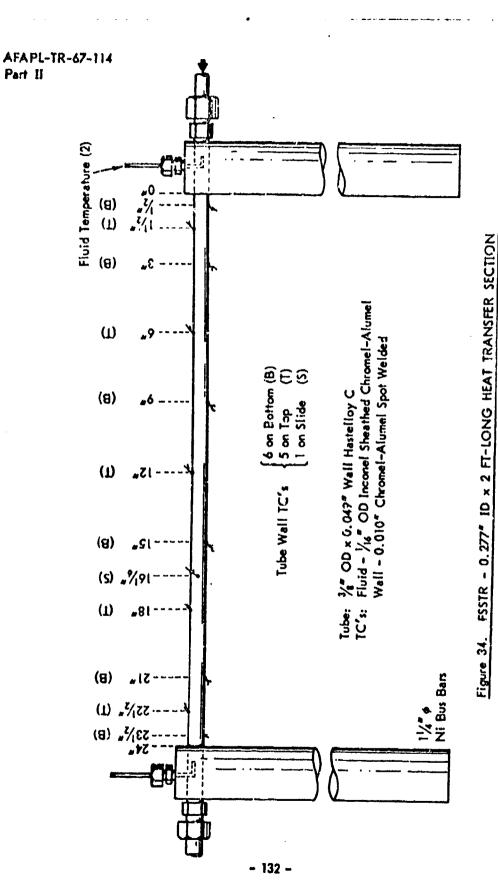
In none of these tests was there any indication of change of heat transfer coefficient that might be attributed to coke deposition. Data from these tests is summarized in Table 49.

In the original design this tube section was constructed with all tube wall thermocouples spot welded to the bottom of the horizontal tube. Series 10018-71 was carried out without any change in this configuration, therefore, temperature differences between top and bottom surfaces of the tube were not measured. Difficulty in obtaining heat balances during these tests led to relocation of the exit fluid thermocouple which in turn gave indication of temperature strata existing in the outlet fluid stream. To determine the extent of this temperature variation along the tube, six additional wall thermocouples were added to the reactor. Five of the new couples were located on top of the tube alternating in axial position with the original six located on the tube bottom and one was positioned on the side of the tube 16-1/2 in. from the inlet end. A sketch of the tube with the revised thermocouple arrangement is given in Figure 34.

Series 10018-75 and 78 were then carried out. In these tests differences of up to 300°F between top and bottom outside wall temperatures were measured. Temperature profiles for three of the runs which were made under similar conditions except for feed temperatures are shown in Figure 35. Data from the 1/2 in. and 23-1/2 in. locations have been deleted from this

Table 49.

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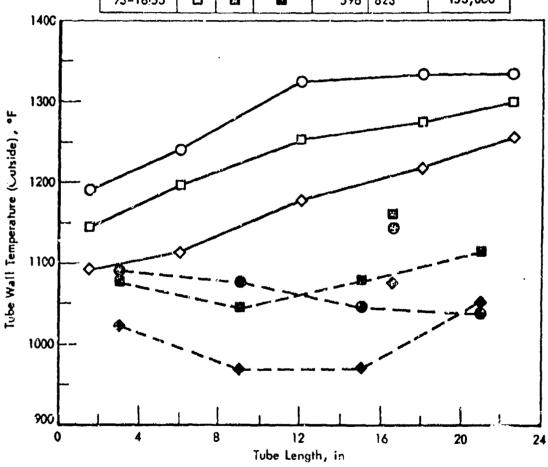


Figure 35. FSSTR - HEAT TRANSFER TO MCH IN EMPTY 0.277" ID x 2-FT LONG
HEAT TRANSFER SECTION: VARIATION OF TUBE WALL TEMPERATURE
WITH THERMOCOUPLE LOCATION

figure as these temperatures are influenced by the proximity of the bus bars. A comparison of the experimental data with several heat transfer correlations is given in another section of this report.

It is assumed that this temperature difference is due to stratification of the flowing fluid rather than being the result of non-uniformity of the wall thickness. A section of the tube from which the reactor was made was measured and a variation of only * 0.001" was found in the nominal 0.049" wall thickness. When time is available this reactor will be operated in an inverted position to see if the hot wall will remain on the top or will move to the bottom with the tube inversion.

Heat losses from the uncompensated 2-ft long reactor section were determined by measuring the temperature drop of a heated N₂ stream passing through the tube in the same manner as was described when it was used as a catalytic reactor. Figure 36 illustrates the measurement for one temperature level. The heat losses determine over the entire temperature range are shown in Figure 28 along with the data obtained using the reactor packed with ratalyst. Note that heat loss to the bus bars ("end" loss) is much less important when the catalyst is no longer present to promote turbulence and increased heat transfer.

Temperature data from Sections I and II of the FSSTR (3/8 in. OD x 10-ft long tubes) taken during lined-out periods of operation when they were being used as preheaters for the 2-ft section are listed in Table 74 in the Appendix.

0.0265" ID x 6" and 4" Heat Transfer Sections

A test set-up designed to operate at higher heat flux conditions than was possible using the larger reactor sections has been assembled and a photograph of the test stand with a 1/16" OD x 6" long heat exchange section in place is shown in Figure 37. The reactor is connected into the FSSTR system and makes use of the feed supply and pressure control system of that unit. Power for resistence heating of the tube is supplied by an 18 KVA variable reactance transformer. The discharge end of the reactor is grounded and is fixed in position to the mounting framework. The upstream end is supported on rails by a movable bracket, and a pulley and weight system is used to keep a constant tension on the tube, moving the bracket as necessary along the rails as the tube expands.

The heat exchange sections used in this rig have all been constructed of 1/16" CD x 0.018" wall type 316 S.S. tube (0.0265" ID) silver soldered to 3/4" diameter copper bus bers. Sketches giving dimensions and thermocouple locations for the five sections used to the present time are given in Figures 38 through 41.

Fluid temperatures are measured in the expanded end fittings before and after the heat exchange section as shown and outside tube wall temperatures are measured by thermocouples which are spot welded to the tube. The wall thermocouple leads are wrapped at least one full turn around the tube before running through ceramic insulation tubes to a junction board. The leads are insulated from direct contact with the tube and each other by a layer of ceramic cement. The rest of the tube has been covered with a

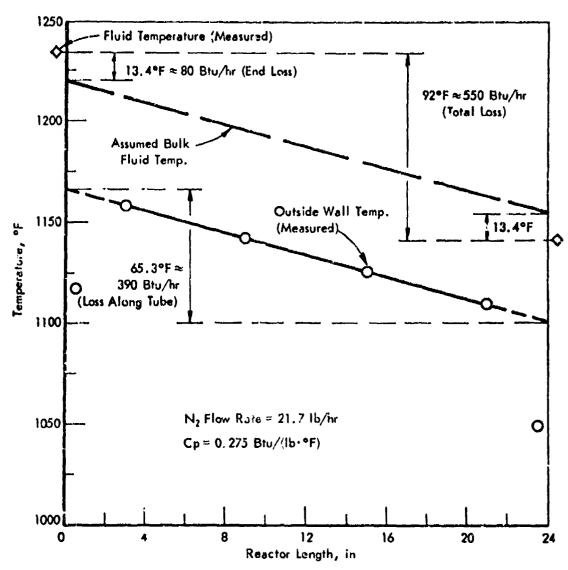


Figure 36. FSSTP: EXAMPLE OF HEAT LOSS MEASUREMENT FROM 2-FT LONG HEAT TRANSFER SECTION

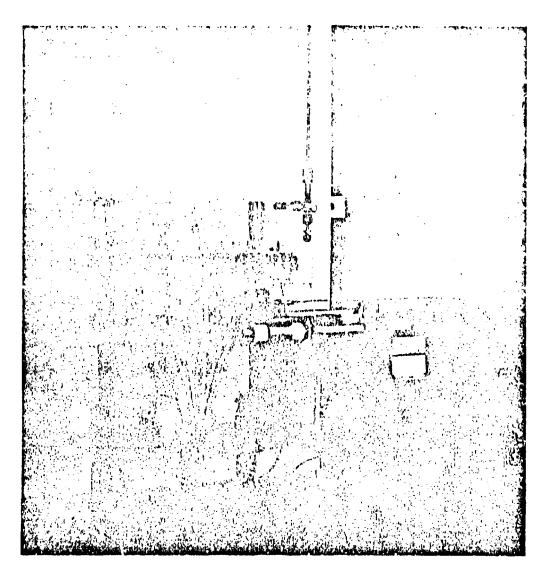
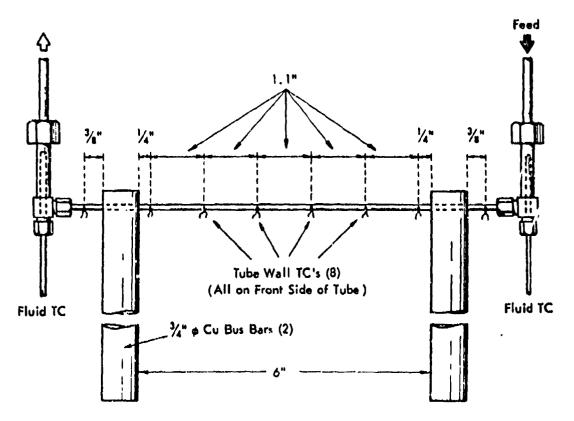


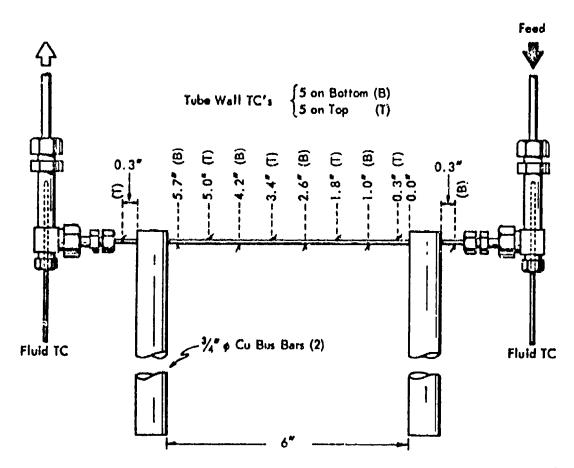
Figure 37. FSSTR - MINIATURE HEAT TRANSFER SECTION TEST STAND



Tube: 1/4" OD x 0.018" Wall Type 315 SS (1/14" Tube 101/4" Long Overall)
Thermocouples: Fluid - 1/16" OD inconel Sheathed Chromel-Alumel

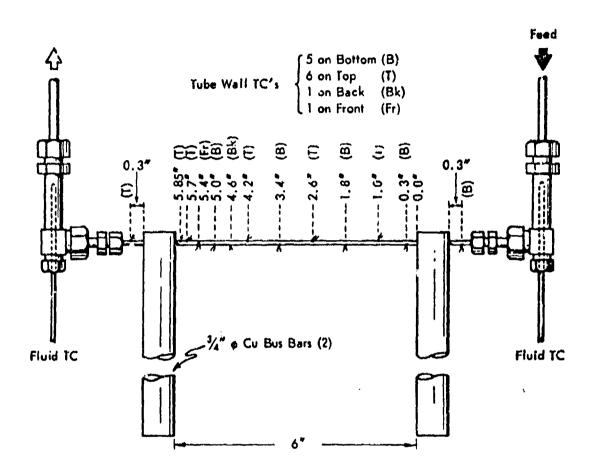
Wall - 0.005" Chromel-Alumel Spot Welded

Figure 38. FSSTR - MINIATURE HEAT TRANSFER SECTION:
REACTOR NO 10018-82



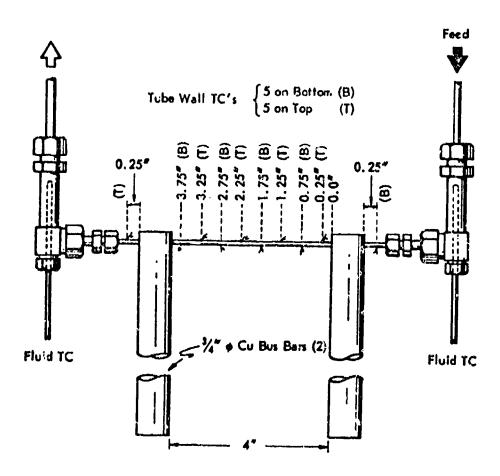
Tube: $\frac{1}{16}$ " OD x 0.018" Wall Type 316 SS ($\frac{1}{16}$ " Tube $\frac{9\frac{1}{2}}{2}$ " Long Overall) Thermocouples: Fluid - $\frac{1}{16}$ " OD Inconel Sheathed Chromel-Alumel Wall - 0.005" Chromel-Alumel Spot Welded

FSSTR - MINIATURE HEAT TRANSFER SECTION: PEACTOR NO. 10018-97



Tube: $\frac{1}{16}$ " OD x 0.018" Wall Type 316 SS ($\frac{1}{16}$ " Tube 9 $\frac{1}{2}$ " Long Overall) Thermocouples: Fluid - $\frac{1}{16}$ " OD Inconel Sheathed Chromel-Alumel Wall - 0.005" Chromel-Alumel Spot Welded

FSSTR - MINIATURE HEAT TRANSFER SECTION: REACTOR NO's. 10018-103 and 110



Tube: $\frac{1}{16}$ OD × 0.018" Wall Type 316 SS ($\frac{1}{16}$ " Tube $7\frac{3}{4}$ " Long Overall) Thermocoupies: Fluid ~ $\frac{1}{16}$ " OD Inconel Sheathed Chromel-Alumel Wall ~ 0.005" Chromel-Alumel Spot Welded

FSSTR - MINIATURE HEAT TRANSFER SECTION: Figure 41. REACTOR NO. 10018-122

similar layer of cement to make heat losses more uniform. Except for runs using the first tube (Reactor 10018-92) the tests have been conducted with ceramic fiber and magnesia block insulation covering the tube and end fittings.

As illustrated in Figure 38 all of the thermocouple junctions were spot welded to the side of the tube when the first miniature section was constructed. The second (Reactor 10018-97) and subsequent tubes were fabricated with alternate couples located on top and bottom of the horizontal tubes (Figures 39 through 41). This was done as tests in the 5/8 in. OD x 2 ft reactor reported above had shown large temperature differences existing between top and bottom of that tube and it was desired to determine if a similar effect could be found in the present small diameter tubes. Data obtained using the second miniature reactor (10018-97) clearly showed that there was indeed a systematic difference between top and bottom wall temperatures. Figure 42 illustrates this difference for a typical run using water feed, where the bottom of the tube was some 20-30°F hotter than the top. Unfortunately, this tube failed before further tests could be made.

The first tests made using the third miniature reactor (Reactor 10018-103) sketched in Figure 40 were made only to clarify these temperature differences. Runs were made under similar conditions, first with the tube in its normal position and then inverted. Figure 43 shows the results of one pair of tests. Contrary to the results from Reactor 10018-97, in this case the top side of the tube was hot when in its normal position. On inverting the tube the hot side moved to the bottom. This indication of nonuniformity in the tube led to further examination of the recovered portion of Reactor 10018-97. The tube was sectioned at three locations (1/2, 3, and 5 in. from the inlet end) and wall thicknesses were measured using a microscope equipped with a micrometer eyepiece. At these three locations the bottom wall was thicker than the top by 0.0021, 0.0015, and 0.006 in., respectively. Nominal wall thickness for this tube was 0.018 inch. Since more heat will be generated in the heavier wall side this explains the observed temperature difference.

For calculation purposes the top and bottom wall temperatures were smoothed as shown in Figure 42, wherever possible, and a uniform wall thickness was assumed. For Series 10018-90 and 94 this could not be done as the thermocouples were all mounted in a single row on the side of the tube. The results for these tests, therefore, are subject to the uncertainty of not knowing if the thermocouples were located where a high, low, or average wall temperature existed.

Heat losses from the miniature heat transfer sections were determined as follows. Loss from the tube wall was found by applying a small amount of power to the section with no fluid flowing. Temperatures were allowed to equilibrate and temperature and power data were recorded. While the tube wall temperature fell off at either end of the section due to heat loss to the bus bars, the profile was flat enough over the central portion of the tube that end losses could be neglected and power input was, therefore, a direct measure of tube wall heat loss at the temperature of that point. This procedure was followed with the tube insulated as was the normal practice when making test runs and also uninsulated to duplicate the conditions for the initial tests using Reactor 10018-82. Loss to the bus bars and end fittings was found by

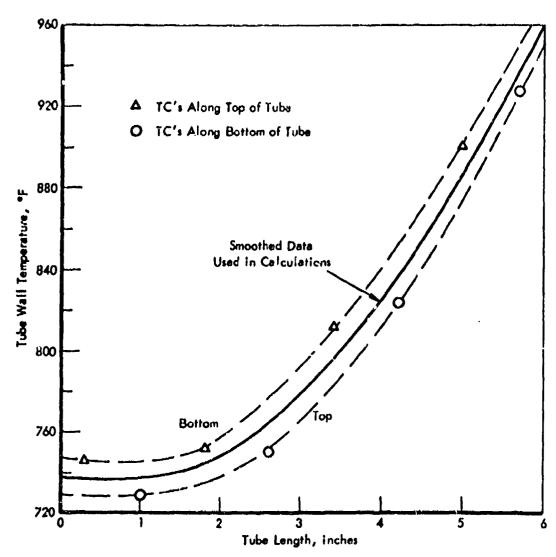


Figure 42. FSSTR: EXAMPLE OF TUBE WALL TEMPERATURE DATA FROM

MINIATURE HEAT TRANSFER SECTION

Run 10018-101-13; 01

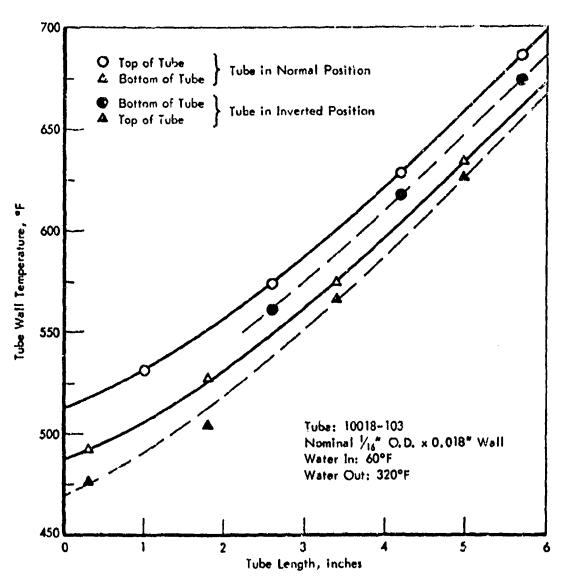


Figure 43. FSSTR: EFFECT OF INVERTING MINIATURE HEAT TRANSFER
SECTION ON TUBE WALL TEMPERATURES

passing a heated feed stream through the tube, measuring the temperature drop and calculating the overall heat loss. The portion of the loss which was directly from the tube wall was determined from the wall temperature and was subtracted from the total to give the loss to the two bus bars and end fittings. This end loss depends on heat transfer between the flowing fluid and the passage through the bus bar and will therefore change with flow rate as well as with temperature of the fluid. Heat loss measurements were therefore made with all fluids used in the test runs and at several different flow rates.

Figure 44 gives the heat loss for the tube wall vs. outside wall temperature and Figure 45 gives the loss to the bus bars as functions of fluid flow rate and temperature.

Test Runs

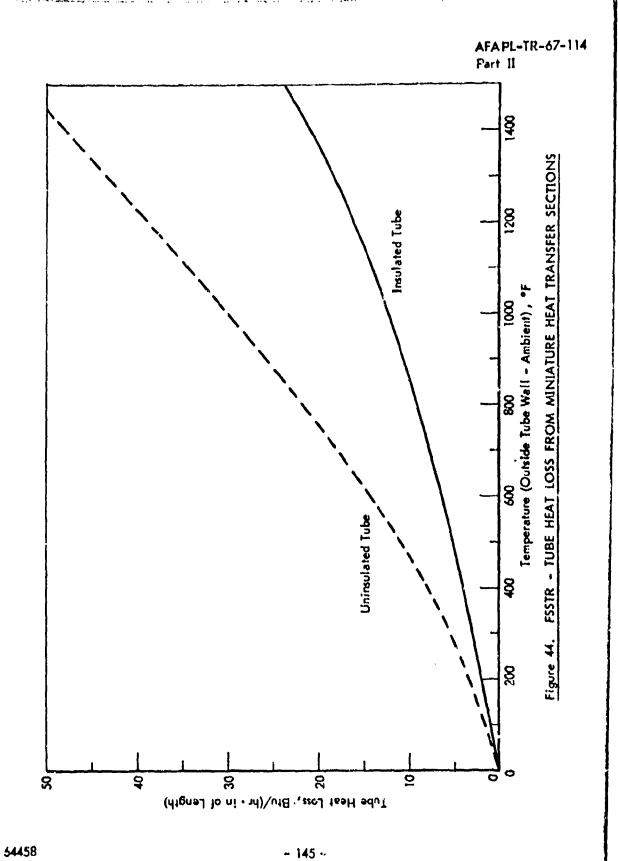
Table 50 presents a brief survey of the operating conditions for which heat transfer data have been obtained using the miniature reactor sections. Tabulated for each test run are the fluid flow rate, inlet and outlet fluid temperature and pressure, heat flux and maximum inside tube wall temperature. Time at temperature is included for the tests using MCH feed where possible deposit formation is of interest.

Two series of tests were conducted using MCH as feed in Reactor 10018-82. The first series (10018-90) was terminated when, at a heat flux of ca 3,500,000 Btu/(hr·ft²), flow and pressure surging became so severe that operation could not continue. Following this test the system was modified so that the product would be cooled and condensed before reaching the pressure control valve. The second test series (10018-94) was made at a higher MCH feed rate and reached a maximum heat flux of ca 4,000,000 Btu/(hr·ft²) without any difficulty. At this point, however, with outlet pressure of 530 psig, bulk fluid temperature of 539°F and maximum inside wall temperature ca 870°F, the tube plugged as a further increase in power was made. The plug was removed by forcing a wire through the tube; however, several of the tube wall thermocouples were damaged and a new tube was constructed before testing was resumed. At this point it was decided to interrupt the MCH tests and continue operations using N₂ and water so heat transfer data could be taken at high heat flux without the added complications of reactor plugging.

Two series of tests (10018-98 and 101) were made using Reactor 10018-97. The first weries, using N_2 at a feed rate of 6.4 lb/hr, was limited to a maximum heat flux of ca 500,000 Btu/(hr·ft²) when wall temperature reached > 1500°F. In the second series (10018-101) water was used as the feed. This test terminated when the tube burned out as the heat flux was raised above 6,000,000 Btu/(hr·ft²).

The third tube (Reactor 10018-103) burned out during its first test series when the power level was increased beyond a heat flux of 1,500,000 Btu/(hr·ft²). Note that the outlet pressure was essentially atmospheric during this test. This reduced the boiling point and caused a lower burn-out heat flux.

less severe conditions were then selected and the next tube (Reactor 10018-110) was used to obtain data while transfering heat to boiling water (Series 10018-116) and to superheated steam (Series 10018-119).



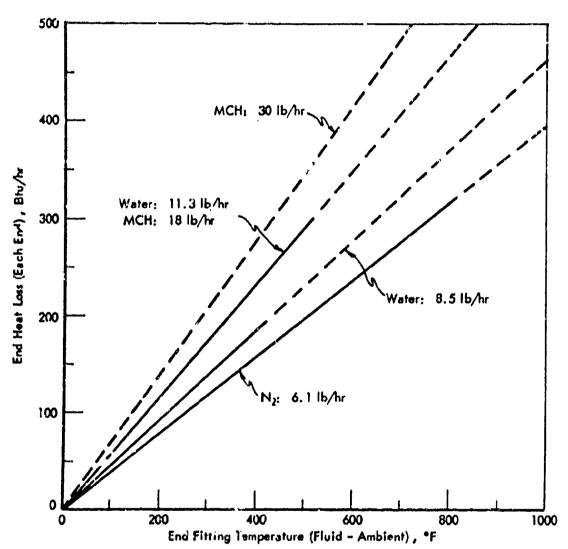


Figure 45. FSSTR - END FITTING HEAT LOSS FROM MINIATURE
HEAT TRANSFER SECTIONS

Table 50. FSSTR - SUMMARY C OPERATING CONDITIONS FOR MINIATURE HEAT TRANSFER SECTIONS

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al fluit temporature and pressure measurer service arrive arrive tents in. But just heated service the last last the first heated service.

The desired maximum 8,000,000 Btu/(hr:ft²) heat flux required the construction of a 4 inch long heat exchange contion (Reactor 10018-122) so that a higher feed rate could be used and a bunn-out heat flux would not be reached. A maximum heat flux of 8,450,000 was attained in Series 10018-166 using this short section.

Tests with MCH were then resumed using Reactor 10018-110. Five series of runs were made at various flow rates as shown, with the power input being limited in most cases when a very sharp pressure drop increase accompanied the final power increase. For the final test using MCH the 4 inch long reactor was reinstalled and used in Series 10018-133 to reach a heat flux of 8.160.000 Btu/(hr·ft²).

More complete tabulations of the data from all of the tests made to date using the miniature heat transfer sections are given in Tables 75 through 88 in the Appendix. Flow, fluid and tube wall temperatures, and pressure readings obtained during the tests are recorded as well as smoothed outside and calculated inside wall temperatures. Heat input values in these tables have been corrected for losses along the tube but no correction has been made for heat loss to the bus bars. This will have to be considered if it is necessary to make an overall heat balance as the outlet temperatures are low as a result of these losses.

Analysis of these results and comparison with several heat transfer correlations is in progress and will be discussed in another section of this report.

While Series 9h was terminated when the reactor plugged there has been no evidence of coke formation during any of the other tests using MCH. It reems possible, therefore, that the plug was not the result of normal operation at the conditions of the run but was formed when a pressure and/or flow upset, perhaps caused by a too abrupt power increase, decreased the fluid flow rate momentarily.

The high fluid and tube wall temperature conditions which would produce coking have been avoided in these tests so that heat transfer data could be accumulated in the region of preation not subject to coking. When this heat transfer data is complete temperature levels will be raised until coke formation is an important factor. It is expected that, with these small tubes and high heat fluxes, even a thin coke deposit will have a considerable effect on heat transfer coefficient and the tube wall temperature will increase rapidly when a coke build-up commences. The possibility of tube failure, during these tests, is to be expected. A further complication will be the necessity for removal of any coke deposit between test runs.

Decalin, STELLLINE H, jet fuel F-71, and possibly methane are expected to be tested as this cooling program continues.

Inside Tube Wall Temperature Calculationsa)

Inside tube wall temperatures (T_WI) were calculated from the measured outside wall temperatures (T_WI) by the following procedure:

The equation for temperature drop across a cylindrical wall with heat being generated uniformly throughout the wall and being transferred both to a fluid stream on the incide and lost to the surroundings on the outer surface is:

$$T_{WO} - T_{WI} = -\frac{(q/A_{1})}{2k_{W}} R_{1} \left[\frac{1 - (R_{1}/R_{0})^{2} + 2 \ln(R_{1}/R_{0})}{1 - (R_{1}/R_{0})^{2}} \right] + \frac{(q_{L}/A_{1})}{k_{W}} R_{1} [\ln(R_{1}/R_{0})]$$
(6)

For the 0.0625 in. 0D \times 0.0265 in. ID tubes used the above equation reduces to:

$$\Delta T_{W} = \frac{(q/A_{1})}{k_{U}(1659)} - \frac{(q_{L}/A_{1})}{k_{W}(1056)}$$
 (7a)

or

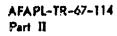
$$\Delta T_{W} = \frac{I^{2}r}{k_{W}} (3.561) - \frac{q_{L}}{k_{W}} (1.638)$$
 (7b)

where

$$q = I^2r$$
 (3.415) Btu/hr $A_1 = 0.0005780 \ rt^2/inch$ tube length.

Resistance and thermal conductivity of type 316 S.S. vary with temperature as shown in Figure 46. Resistance values for the tubes used were determined by measuring resistance at ambient temperature and correcting for temperature variation by applying ratios of resistance between ambient and elevated average wall temperatures obtained from Figure 46. The following equations show the resulting resistance and thermal conductivity for a typical tube with resistance equal to 0.0120 chm/inch of length at 68°F.

- a) Symbols used in this section are as follows:
 - A tube surface area, ft2/unit length; A inside, Ao outside
 - h heat transfer coefficient, Btu/(hr.ft2.°F)
 - I current, amperes
 - k, thermal conductivity of tube material, Btu/(hr.ft.°F)
 - q heat generated in wall, Btu/hr
 - q, heat lost to air, Btu/hr
 - R tube radius, ft; Ri inside, Ro outside
 - r tube resistance, ohm/unit length
 - T temperature, °F; T_{WI} = inside wall, T_{WO} = outside wall, T_{WA} = average wall, T_{r} = bulk fluid, ΔT_{W} = T_{WO} T_{WI} .



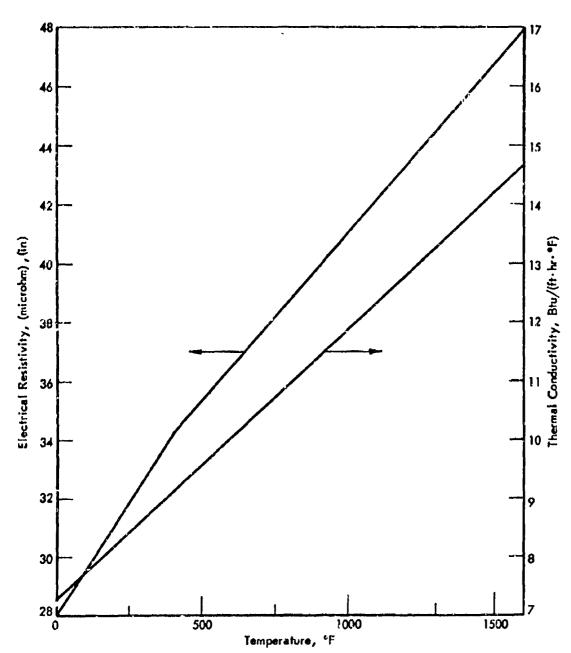


Figure 46. FSSTR - THERMAL CONDUCTIVITY AND ELECTRICAL RESISTIVITY
USED FOR TYPE 316 SS MINIATURE HEAT TRANSFER SECTIONS

From 0-400°F, $r = 0.01156(1 + 0.0005597 T_{Wa})$ (8a)

From 400-1500°F, $r = 0.01234(1 + 0.0003729 T_{MA})$ (8b)

From 0-1500°F, $k_{\rm w} = 7.33(1 + 0.0006248 T_{\rm WA})^{\rm mass}$ (9)

On substituting these expressions in Equation (7a)

From 0-400°F.

$$\Delta T_{W} = I^{2}(0.005616) \frac{(1 + 0.0005597 T_{WA})}{(1 + 0.0006248 T_{WA})} - q_{L} \frac{(0.2255)}{(1 + 0.0006248 T_{WA})}$$
(104)

From 400-1500°F,

$$\Delta T_{W} = I^{2}(0.005995) \frac{(1 + 0.0003729 T_{WA})}{(1 + 0.0006248 T_{WA})} - q_{L} \frac{(0.2235)}{(1 + 0.0006248 T_{WA})}$$
 (10b)

These equations were solved for $\Delta T_{\mathbf{W}}$ as follows:

A parabolic temperature gradient through the wall was assumed such that

$$T_{WA} = T_{WO} - 0.353 \Delta T_{W}$$

Making this substitution into the first term of Equation (10a) or (10b) and using the measured values of I and T_{WO} an approximate value for ΔT_W was obtained which in turn gave a close approximation of T_{WA} . The heat loss correction to ΔT_W was then determined by using this approximate value of T_{WA} along with the value of q_L from Figure 44 in the second (heat loss) term of Equations (10a) or (10b). Since this correction to ΔT_W was very small (2°F or less) which resulted in < 1°F change in the original approximation of T_{WA} it was not necessary to repeat the calculation with modified T_{WA} values.

Heat flux and total heat to the fluid at intermediate points along the tube were determined as follows:

Actual heat flux to the fluid is $(q/A_1) - (q_1/A_1)$. Heat generated in the tube per unit length (q) can be determined using the measured current (I) and the approximate resistance (r) from Equation (8a) or (8b) evaluated at the average tube wall temperature at that point (T_{ijk}) . For these tubes the inside surface area per unit length (A_1) is 0.0005780 ft²/inch of tube length. Total heat transferred to the fluid up to any point along the tube was determined simply by averaging the heat flux up to that point and multiplying this average by the inside surface area up to that point.

Model of a Packed Bod Reactor

Influence of Physical Properties

In the packed bed reactor program, physical properties for MCH dehydrogenation (compressibility factor, thermal conductivity and viscosity) were changed to improved values. These were then varied separately to determine the sensitivity of calculated results to a change in each property. Values of the properties and the results are shown in Table 13 along with corresponding experimental measurements. The properties for Case A were determined at an average pressure and remperature for the experimental runs. The compressibility factor in Case B is in the range of low MCH conversion. However, it increases rapidly with increasing conversion and is approximately 1.0 at moderate and high conversions. Hence, 1.0 is a reasonable value in most calculations. The thermal conductivity in Case C and the viscosity in Case D were determined at the highest pressure and temperature encountered in the experiments.

Variations in the thermal conductivity and the viscosity had negligible effect on the calculated results. A change in the compressibility factor had some effect on the outlet temperature and conversion; however, the changes are comparable to differences between the calculated and experimental results. The only significant change in the results is the increase in outlet pressure due to a decrease in the compressibility factor. For the four runs shown, the ratio of the pressure drop for Case B to that for Case A is approximately 0.8, alightly less than the ratio of the compressibility factors for the two cases. In general, the agreement between calculated and experimental results is good for a compressibility factor close to 1.0, which is to be expected since it rises rapidly to 1.0 as MCH reacts.

The above results show that the use of constant properties in the packed bed reactor program is justified, since the sensitivity of these results to property changes in a single phase is small.

Reaction Kinetics for MCH Dehydrogenation

Kinetic Parameters From FSSTR Data

Further calculations with the packed sed reactor program have improved the values of kinetic parameters for NCH dehydrogenation. The kinetic model for MCH dehydrogenation is similar to the desorption-controlling model proposed by Sinfelt et al, 53 and is the same as rate expressions used in a previous report: 18)

$$r_{MCH} = \frac{(1-c)k_1k_2c_{MCH}}{1+k_2c_{MCH}} \left[1 - \frac{p_{TOL}^2H_2^3}{p_{MCH}^Keq}\right]$$
(11)

where

r_MCH = rate of MCH dehydrogenation

e = void fraction

 $k_1 = A_1 \exp(B_1/RT)$

Table 51. EFFECT OF CHANGES IN PHYSIC/L PROPERTIES
ON MICH DEHYDROGEN/TION

Run Number	140- 1540	198- 1420	5- 1620	50- 1310
G, lb/ft²-hr To, °F Po, psig q, Btu/ft²-hr	50400 1090 890 27500	36800 894 407 13300	33200 900 903 24100	1.54100 900 885 35900
	Cutlet Ten	pereture,	•F	
Experiment Calculation,	1009	632	858	745
A B C D	1005 995 1005 1005	657 707 697 657	833 836 833 833	755 755 755 756
	Outlet Pre	essure, poi	lg	
Experiment Calculation, Case	#46	21;	790	671
A B C D	478 572 478 473	230 291 230 225	792 813 • 732 792	668 710 668 658
	fiCH Conv	ersion, 🎋		
Emperiment Calculation, Case	94	1414	62	21.3
Case A B C D	94.4 95.5 94.4 94.4	44.1 43.5 44.1 44.1	64.4 64.3 64.4 64.4	20.1 20.2 20.1 20.1
		Prope	rties	
Саво	z	k, Btu/ft-		μ, 15/ft-hr
A B C D	0.97 0.85 0.97 0.97	0.05 0.05 0.05 0.05	143 170	0.0417 0.0417 0.0417 0.0430

 $k_2 = A_2 \exp(B_2/RT)$

A1, A2 - pro-exponential factors

B₁ = activation energy for toluene desorption

B2 - energy of reaction for MCH to adsorbed toluene

R - universal gas constant

T - absolute temperature

c = concentration of MCH

Prof. = partial pressure of toluene

Pu = partial pressure of hydrogen

PMCH = partial pressure of MCH

 $K_{eq} = A_3 \exp (B_3/RT)$

An = 4.0 x 1020 atm2

 $B_a = -92.500 \text{ Rtu/lb mole}$

The latest calculations utilized experimental results which were obtained on a 2-ft section in the FSSTR and were not previously available, along with earlier data obtained on 10 ft sections in the FSSTR. Data and results for selected runs are shown in Table 52. Reasonable agreement between calculation and experiment was obtained by varying one of the kinetic parameters, A₁, from the value used in previous calculations.

The improved kinetic parameters are

 $A_1 = 1.2 \times 10^{13} \text{ lb mole/ft}^3\text{-hr}$

 $A_2 = 4.5 \times 10^{-8} \text{ ft}^3/\text{1b mole}$

 $B_1 = -59,000$ Btu/lb mole

 $B_{\mathbf{p}} = 54,000 \text{ Btu/lb mole}$

The values of B_1 and B_2 are approximately the same as those determined by Sinfelt, but A_1 has a much larger value, which is indicative of a more active catalyst.

Better agreement between calculation and experiment also resulted when the more accurate reactor lengths shown in <u>Table 52</u> were used instead of the approximate values of 2 and 10 feet.

Figures 47 and 48 show calculated and experimental results for two MCH dehydrogenation runs in the 2-ft section of the FSSTR. The agreement is good for the outlet pressure and conversions. There is some disagreement between the calculated and experimental temperature profiles. This may be due to different values of the heat transfer coefficient in the calculations and the experiment. In the calculations, the heat transfer coefficient is constant and yields an almost constant temperature difference between the fluid and the wall. In the real case, the number of moles of gas increases. Hence, the linear velocity and the heat transfer coefficient increase, causing the temperature difference between the fluid and the wall to decrease,

Table 52. CALCULATED AND EXPERIMENTAL RESULTS FOR MOR DEHYDROGENATION IN THE PACKED BED RELOTOR

			Date						
Run Munter	146- 1340	148- 1540	198- 1420	1620	1400	50-	50- 1510	62- 1300	Se- 1415
Reactor diameter, in.	0.277	0.277	0.552	0.652	0.652	0.277	0.277	0.277	0.277
Reactor length, ft	9.31	10.9	9.55	9.55	9.55	2.04	2.0t	2.0t	2.2
Nuid mass flux, lb/ft2 hr	35700	60400	36800	33200	35700	154100	154100	60000	6000
Wall heat flux, Stu/it2 hr	11100	36000	13900	25200	52350	145400	311300	25050	156000
Nuid enthalpy change, $ heta tu / t b$	514	ફૂ	566	534	1003	327	71.5	171	88
Inlet pressure, psig	1430	&	£3	903	88	385	885	168	889
Inlet temperature, *F	797	1090	1 60	8	905	88	910	901	895
		Calcu	Calculated Results	esilts					
Outlet presenre, psig	243	684	942	797	169	596	452	857	830
Outlet temperature, *F	72	985	695	832	1025	827	1069	766	1055
MCH conversion,	61.9	96.1	44.1	64.3	98.0	10.1	8.8	29.1	8.48
		Experi	mentel	Experimental Results					
Outlet pressure, psig	767	844	ध्य	790	919	1 8	991	855	825
Outlet temperature, 'F	7775	1000	682	838	1034	831	101	770	101
MCH conversion, 🕹	59	94	14	62	27	39.1	65.9	28.7	86

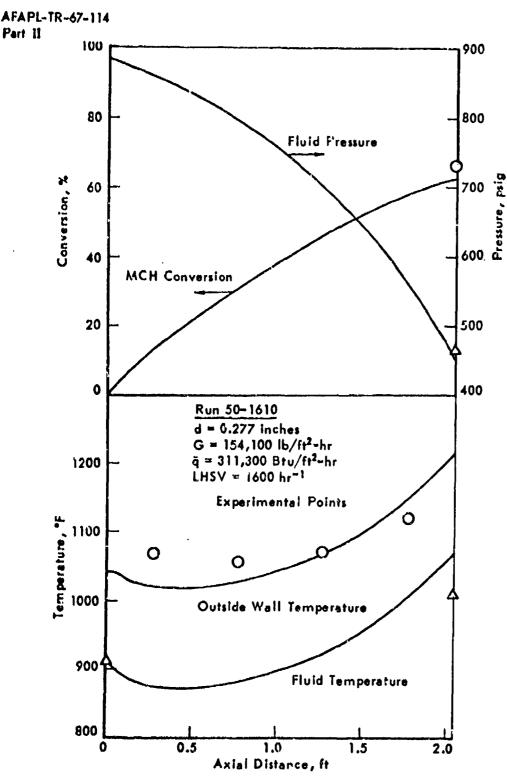


Figure 47. CALCULATED PROFILES FOR MCH DEHYDROGENATION
IN THE FSSTR FROM FSSTR KINETIC DATA
- 156 -

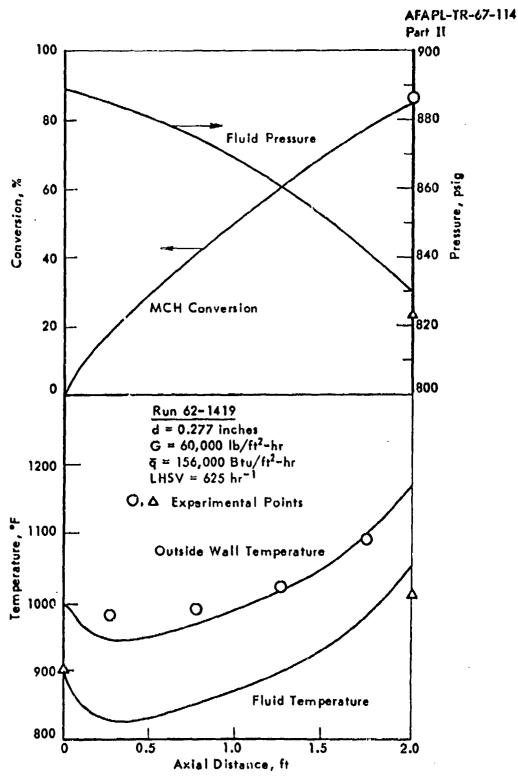


Figure 48. CALCULATED PROFILES FOR MCH DEHYDROGENATION IN THE FSSTR FROM FSSTR KINETIC DATA

as indicated by the decreasing difference between the curve for the fluid temperature and the experimental points for the wall temperature.

Kinetic Parameters From Bench-Scale Data

In order to determine the feasibility of using bench-scale data to calculate kinetic parameters for Decalin dehydrogenation, data obtained on the bench-scale reactor have been used to determine the kinetic parameters for MCH dehydrogenation for comparison with the results calculated from the FSSTR data. The bench-scale data were evaluated for a desorption-controlling kinetic model by a nonlinear estimation technique.

The kinetic model is a simplified version of Equation (11). Since equilibrium was not important in any bench-scale runs, the binomial factor was dropped and Equation (11) reduced to

$$r_{MCH} = \frac{(1-\epsilon)k_1k_2c_{MCH}}{1+k_2c_{MCH}}$$
 (12)

The model was developed for an integral reactor, since high conversions occurred on several runs. Isothermal conditions were assumed to simplify the integration. Steady-state conditions were assumed for the continuity equation:

$$\nabla \cdot \underline{\mathbf{n}}_{MCH} = \mathbf{r}_{MCH} \tag{15}$$

where \underline{N}_{MCH} = molar flux of NCH.

The MCH flux is assumed to be due entirely to convection in the axial direction. Hence

$$\frac{d(c_{MCH}v_2)}{dz} = r_{MCH}$$
 (14)

where v_z = average velocity in axial direction

z = axial distance.

The MCH concentration is related to the molar concentration of the gas by the reaction stoichiometry:

$$c_{MCH} = c \left(\frac{1-g}{1+3g} \right) \tag{15}$$

where c = molar concentration of the gas

g = fractional conversion for dehydrogenation of pure MCH.

The stoichiometry of the reaction also relates the average velocity at any time to the initial velocity. The compressibility factor is assumed to be unity, which is valid for moderate and high conversions and reasonable for low conversions. Hence

$$v_z = v_{z0}(1 + 3g)$$
 (16)

where v_{2O} = initial average velocity.

Also

$$e = \frac{P}{RT} \tag{17}$$

where P = absolute pressure.

By substitution of Equations (12), (15), and (16) the continuity equation is transformed to

$$\operatorname{ev}_{20} \frac{\operatorname{dg}}{\operatorname{dz}} = \frac{(1-\epsilon)k_1k_2c(1-g)}{1+3g+k_2c(1-g)}$$
 (18)

Integration along the exial direction from z = 0 yields

$$\frac{(1-\epsilon)k_1k_2z}{v_{Z_0}} = \int_0^g \left(k_2c - 3 + \frac{4}{1-\epsilon}\right) dg$$

$$\frac{(1-\epsilon)k_1k_2z}{v_{Z_0}} = (k_2c - 3)g - 4 \ln(1-g)$$
(19)

Introduction of the mass flux for the initial velocity, reactor volume for axial distance, and Equation (17) for concentration yields

$$\frac{k_1 k_2 PVM_0(1-\epsilon)}{RT GA} = \left(\frac{k_2 P}{RT} - 3\right) g - 4 \ln(1-g)$$
 (20)

where V = volume of tubular reactor

Mo = molecular weight of feed

G = mass flux

= cross-sectional area of tubular reactor.

Equation (20) relates the reactor volume to the MCH conversion for given values of the mass flux, pressure, and temperature.

The kinetic parameters in the rate equation were determined by nonlinear estimation after rearrangement of Equation (16) to

$$V = \frac{GA}{ib(1-\epsilon)} \left\{ \frac{g}{k_1} - \frac{gT}{k_1 k_2 P} \left[3g + 4 \ln(1-g) \right] \right\}$$
 (21)

The reactor volume was taken as the response function with mass flux, pressure, temperature, and conversion as independent variables. The error sum of squares for the reactor volume was minimized to yield the following kinetic parameters:

 $A_1 = 1.8 \times 10^{11} \text{ lb-mole/ft}^3\text{-hr}$ $A_2 = 3.6 \times 10^{-4} \text{ ft}^3/\text{lb-mole}$ $B_1 = -48800 \text{ Btu/lb-mole}$ $B_2 = 31700 \text{ Btu/lb-mole}$

Attempts to evaluate a confidence region for the parameters were unsuccessful. These parameters are significantly different from those in the previous section. B_1 and B_2 are less, and since these are exponents A_1 and A_2 are quite different.

Comparison of Results

MCH dehydrogenation rates were determined for kinetic data from two different sources and are shown in Table 55. Rates based on data from the bench-scale reactor and FSSTR are generally similar although the former data predict slightly higher rates. However, these rates are 10 to 100 times faster than the corresponding rates based on Sinfelt's work. Rates predicted from Sinfelt's data are questionable at 800°K, since these were determined by extrapolation from data at 588-645°K.

Table	53.	DEHYDROGUNATION	RATES	FOR	PURE	MCH

	Re	action R	ate, g-m	ole/hr-g	Catalys	t
Source Kinetic Data	€00°K	(520°F)	700°K	(800°F)	800°K	(980°F)
	20 atm	50 atm	20 atm	50 aim	20 atm	50 atm
Pench-Scale Resctor	0.29	0.70	5.5	6,7	27.	47.
FSSTR	0.15	0.15	5.6	6.0	18.	39 •
Sinfel; et al7)	0.017	0.019	0.11	0,23	(0.16)	(0.39)

The kinetic parameters determined from bench-scale data were used in the packed-bed reactor program. The conditions shown in Table 54 for various experimental runs were used as data for the calculations. The predicted results are compared with experimental measurements in Tables 55 to 57. The calculated conversions in Table 55 are slightly higher than the experimental conversions. In Tables 56 and 57 most of predicted and experimental conversions agree with each other. Outlet temperatures for each run show the same degree of agreement as the conversions, since energy balances on the reacting fluid were used to determine the heat fluxes.

Figures 49 and 50 show calculated and experimental results for MCH dehydrogenation in the 2-ft section of the FSSTR. These are the same runs as shown in Figures 47 and 48. The agreement is good for fluid temperature, pressure, and conversion. There is some disagreement between the calculated and experimental wall temperature with the greatest difference being approximately 30°F near the reactor inlet. Other runs in the 2-ft reactor had good agreement except for Runs 50-1630 and 60-1336. The difference between the calculated and observed results in the former run is due to significant catalyst deactivation. However no apparent cause accounts for the disagreement in Run 60-1336.

Table 54. DATA FOR THE PACKED BED PROGRAM PREDICTIONS OF MCH DEHYDROGENATION RUNS

Run No.	Mass Flux of Fluid, lb/ft ² -hr	Heat Flux to Fluid, Btu/ft2-hr	Inler Pressure, paig	Inlet Temp, *F
Cataly	et Bed Dimens	ions: 0.277-i	n. D x 9.81-1	rt L
120-1400 123-1505 144-1330 144-1530 144-1530 144-1640 146-1340 146-1500 146-1620 143-1320 148-1540	59,500 71,700 37,600 37,500 37,500 37,500 36,700 35,800 35,800 60,400	35,800 38,200 0 9,600 21,500 19,100 11,200 7,300 19,700 28,600 27,900	890 887 626 630 633 434 430 890 891 886 890	925 935 782 790 788 786 797 801 793 935 1090
Cataly	st Bed Dimens	ions: 0.652-1	n. D x 9.55-1	tL
198-1300 198-1420 5-1200 5-1320 5-1620 9-1300 9-1400	35,500 36,800 17,200 17,200 33,200 36,700 35,700	15,160 13,400 12,400 23,900 25,400 35,700 52,200	911 487 901 903 903 885 886	895 894 898 900 900 904 905
Cataly	st Bed Dimen	sions: 0.277-	in. D x 2.04	ft L
50-1210 50-1310 50-1400 50-1510 50-1540 50-1610 50-1630 60-1336 62-1300 62-1400 62-1419 52-1650	154,100 154,100 154,100 154,100 154,100 154,100 154,100 154,100 154,100 60,000 60,000 60,000 60,000	-5,000 37,000 83,000 139,500 202,400 253,700 306,200 338,100 -5,000 267,300 -3,400 28,800 115,100 152,600 30,200	885 885 885 885 885 885 885 891 892 892 891 899 891	999 900 891 893 908 910 899 900 901 901 895 898

Table 55. EXPERIMENTAL AND PREDICTED RESULTS FOR MCH CEHYDROGENATION IN THE FOSTR (0.277-IN. D x 9.81-FT L)

Run No.	Conv.,	Outlet Pressure,	Outlet Temp,		ine Flui ure, °F,	
		psig	•F	2.5 ft	5.0 ft	7.5 st
120-1400	96.	502	1030	795	850	830
Prediction	98.2	508	1019	785	850	880
123-1505	91.	125	1040	800	829	873
Prediction	95.3	194	988	783	821	860
144-1330	9.	570	637	659	650	646
Prediction	10.5	555	621	635	ავი	625
144-1430	49.	524	784	712	738	751
Prediction	50.5	518	765	693	724	748
144-1530	93.	469	963	751	790	835
Prediction	95.8	467	928	737	786	834
144-1640	85.	144	915	728	754	734
Prediction	89.5	128	861	707	743	775
146-1340	59.	192	775	707	719	734
Prediction	62.4	231	732	681	707	722
146-1500	40.	826	800	729	751	771
Prediction	41.3	826	783	707	739	763
146-1520	90.	773	958	772	812	862
Prediction	92.4	790	929	757	812	858
148-1320	88.	516	947	791.	&1	858
Prediction	91.5	530	904	775	814	848
148-1540	94.	443	1009	805	838	882
Prediction	97.1	467	971	811	835	875

Table 56. EXPERIMENTAL AND PREDICTED RESULTS FOR MCH

DEHYDROCENATION IN THE FSSTR

(0.652-IN. D x 9.55-FT L)

THE STATE OF THE S

Run No.	Conv.,	Outlet Pressure,	Outlet Temp,		ine Flui ure, °F,	
	٠,	paig	F	2.5 ft	5.0 ft	7.5 ft
193-1300	43.	მ 0 9	782	732	750	761.
Prediction	42.3	მ 0 უ	783	725	749	766
198-1420	44.	219	682	687	687	684
Prediction	43.7	215	586	678	688	690
5-1200	64.	871	825	746	777	800
Prediction	63.4	872	835	742	778	805
5-1320	98.	860	1013	773	821;	872
Prediction	98.4	863	1007	773	826	879
5-1620	62.	790	838	749	777	798
Prediction	62.8	789	828	743	777	802
9-1300	77.	720	872	758	792	817
Prediction	78.0	720	353	753	793	821
9-1400	97•	6 76	1034	777	823	866
Prediction	98.5	678	1014	772	823	870

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Table 57. EXPERIMENTAL AND PREDICTED RESULTS FOR MCH

DESTURIORIESTION IN THE INSTER

(0.277-IN. D x 2.04-FT L)

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Fun No.	Cony.,	Outlet Presoure, paig	Outlet Temp,	Outside Wall Temperature, *F, at			
				0.27 ft	0.77 ft	1.27 ft	1.77 ft
50-1210	15.2	700	688	801	738	715	703
Prediction	15.6	679	683	800	733	702	687
50-1310	21.5	671	745	839	787	772	771.
Prediction	21.8	647	738	8 31	778	761	757
50-1400	28.5	645	789	885	846	841	846
Prediction	28.9	623	733	862	825	818	820
50-1430	39.1	604	831	937	906	907	919
Prediction	39.1	585	831	903	880	882	990
50-1510	51.2	548	880	987	965	970	989
Prediction	50.7	532	887	952	940	95 0	968
50-1540	59.6	505	935	1025	1007	1016	1046
Prediction	59.0	485	944	990	986	1004	1036
50-1610	65.9	466	1011	1061	1048	1063	1108
Prediction	66.2	432	1014	1.027	1033	1061	1112
50-1630	65.3	432	1101	1098	1097	1119	1200
Prediction	69.3	405	1058	1046	1060	1095	1160
60-1300	14.7	709	696	802	739	713	700
Prediction	15.5	631	684	300	732	702	687
60-1336	63.	505	926	1023	1011	1027	1050
Prediction	60.8	488	953	993	998	1018	1053
62-1130	13.9	865	699	756	711	702	70).
Prediction	14.8	862	686	747	695	688	685
62-1500	28.7	855	770	8აგ	782	790	801
Prediction		856	766	791	762	771	782
62-1400	72.3	835	901	931	932	957	990
Prediction	72.0	842	905	903	909	939	971
62-1419	86.	823	1002	981	990	1022	1089
Prediction	87.0	828		949	966	1007	1066
62-1650	28.0	854	788	815	787	793	809
Prediction	29.5	856	768	792	764	774	785

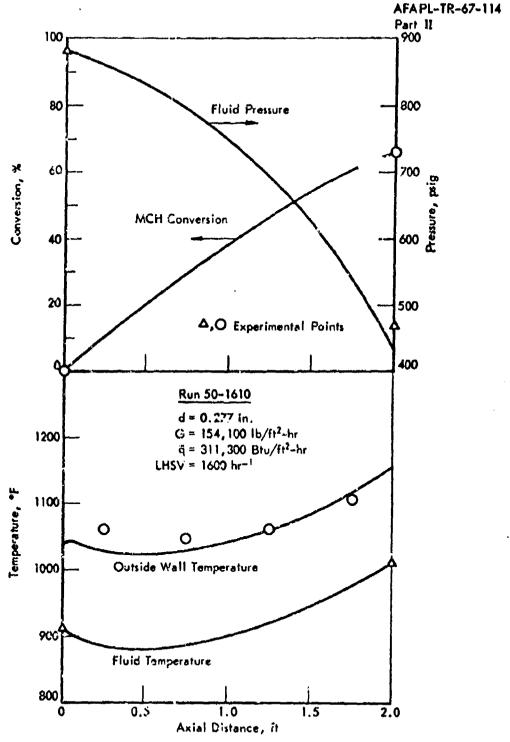
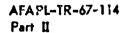


Figure 49. CALCULATED PROFILES FOR MCH DEHYDROGENATION
IN THE FSSTR FROM BENCH-SCALE KINETIC DATA



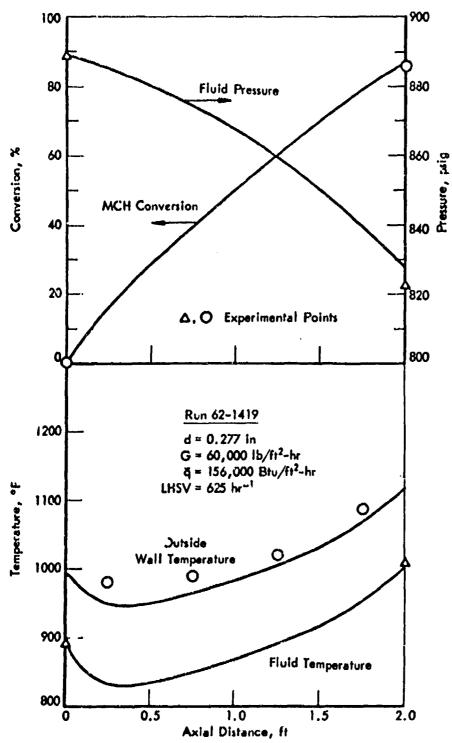


Figure 50. CALCULATED PROFILES FOR MCH DEHYDROGENATION
IN THE F3STR FROM BENCH-SCALE KINETIC DATA

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Figures 57 to 60 generally show slight differences in the predictions based on the two sets of data. The kinetic parameters based on the bench-scale data do predict slightly higher conversion and pressure drop and slightly lower temperature, which becomes appreciably lower at the reactor exit, than the parameters based on FSSTR data. They also predict a temperature profile that is a slightly better fit of the experimental data and has a curvature more like the experimental profile. Because of this the bench-scale reactor should be suitable for collecting kinetic data, the kinetic parameters calculated from bench-scale data for MCH dehydrogenation should be reasonable, and available bench-scale data can be used to determine a kinetic scheme for Decalin dehydrogenation.

Reaction Kinetics for Decalin Dehydrogenation

Decalin-Tetralin-Naphthalene Equilibria

... computer program was developed and used to calculate equilibrium compositions of the Decalin tetralin-naphthalene system at various conditions. Since the program will ultimately be used in kinetic calculations for Decalin dehydrogenation, it is presently restricted to mixtures that result from the dehydrogenation of pure Decalin.

The independent chemical reactions used for the system are

trans-
$$+ 3H_2$$
 (23)

Equilibrium constants for the above reactions are:

$$k_1 = \frac{\gamma_{tD} \cdot tD}{\gamma_{cD} \cdot cD} = k_{\gamma_1} \cdot \frac{g_1 - g_2}{1 - g_1}$$
 (25)

$$k_{2} = \frac{\gamma_{T}\gamma_{H_{2}}^{3} \chi_{T}P_{H_{2}}^{3}}{\gamma_{t}D\chi_{t}D} = k\gamma_{2}P_{H_{2}}^{3} \left(\frac{g_{2} - g_{3}}{g_{1} - g_{2}}\right)$$
(26)

$$k_3 = \frac{\gamma_N \gamma_{H_2}^2 X_N P_{H_2}^2}{\gamma_{TXT}} = k \gamma_3 P_{H_2}^2 \left(\frac{g_3}{g_2 - g_3} \right)$$
 (27)

where

ki = equilibrium constant of reaction i

kyi = equilibrium constant for the activity coefficients of reaction i AFAPL-TR-67-114 Part II

gi = equilibrium conversion of reaction i

7j = activity coefficient of chemical species j

X5 " mole fraction of chemical species j

P_j = partial pressure of chemical species j

and chemical species are represented by the suboripts

oD = cis-Decalin

tD = trans.Decalin

T = tetralin

N = naphthalene

He - hydrogen

The partial pressure of hydrogen is related to conversion by

$$P_{H_{2}} = \left(\frac{3g_{2} + 2g_{3}}{1 + 3g_{2} + 2g_{3}}\right)P \tag{28}$$

where

P = total pressure

Value of the equilibrium constants are determined from a least squares fit of $\det^{21}(2P)$ by

$$\log K_1 = a_1 + \frac{b_1}{T}$$
 (29)

where

ai, bi = constants

T = absolute temperature

Equations (25) to (28) are solved by iteration to determine the reaction conversions at any given temperature. The iteration logic follows:

- 1. Assumed values are assigned to the reaction conversions.
- 2. Mole fractions are calculated for each chemical specieu.
- 3. The fugacity of the mixture is calculated by Pitzer and Curl's equation of state for the second virial coefficient. 54) 55)
- 4. Activity coefficients are determined for each species by the method of Gamson and Watson.

- 5. Equilibrium constants for the activity coefficients are calculated.
 - 6. The hydrogen partial pressure is calculated by Equation (28).
- 7. Equations (25) to (27) are solved simultaneously to yield the reaction conversions.

The conversions calculated in step (7) are assumed as the starting point for the following iteration. The iterations are terminated when the calculated conversions match the assumed conversions. Rapid convergence was achieved when the starting point was the complete conversion of initially pure Decalin to naphthalene and hydrogen. If feeds other than pure Decalin are used, the same starting point and logic should likewise result in rapid convergence to a solve, on.

Equilibrium compositions and reaction conversions are shown in Figures 16 and 51 as functions of temperature at various pressures. The curves are based on equilibrium mixtures that result when pure Decalin dehydrogenates to tetralin, naphthalene, and hydrogen. At 10 atm, Decalin has reacted completely at 740°F with 10% converted to tetralin and 90% to naphthalene. At 50 atm the 90% conversion to naphthalene occurs at 870°F. Hence, over most of the conditions to be expected in the FSSTR (700-1100°F, 10-60 atm), the equilibrium conversion of Decalin is almost complete, and the conversion to naphthalene is equilibrium limited only at the lower temperatures and higher pressures.

Kinetic Parameters From Bench-Scale Data

Present work on Decalin dehydrogenation consists of evaluating experimental data from bench-scale studies to determine a kinetic model for use in the packed bed reactor program. The data are being used to select the best form of the rate expression and to estimate kinetic parameters. Later FSSTR data will be used to confirm the model and improve the values of the parameters.

In the reaction model, cis- and trans-Decalin are considered as separate chemical species. Both isomerize to each other and dehydrogenate to tetralin, which dehydrogenates to naphthalene. The reverse hydrogenation steps will be included if the kinetic model is significantly improved. Basically the model has one isomerization and two dehydrogenation steps. In the kinetic scheme three conversions are associated with these steps. In the analysis of the bench-scale data, reaction rates for the three steps will be integrated numerically for each run to obtain conversions. Least squares analysis will be used on the conversions to optimize kinetic parameters and then select the best kinetic model.

Model of a Regenerative Heat Exchanger for Missile Application

Model Development

U FOREST

One of the rear term applications of Air Force programs on vaporizing and endothermic fuels involves utilization of supersonic combustion ramjet engines for powering missiles. Present plans contemplate using only

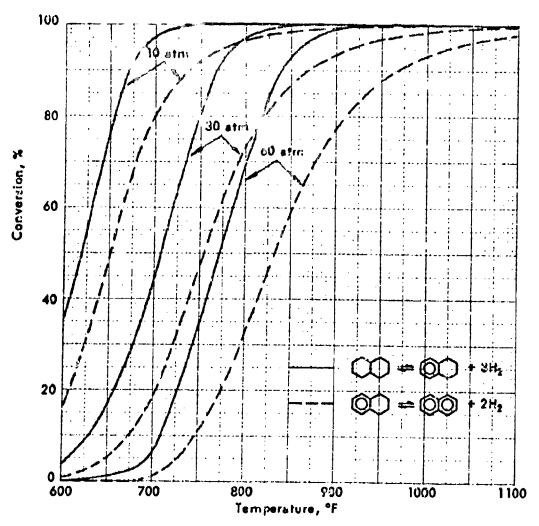


Figure 51. EQUILIBRIUM CONVERSIONS OF THE DECALIN-TETRALIN-NAPHTHALENE SYSTEM

the latent and sensible heat capacity of the fuel for cooling the engine. Under the current contract the behavior of candidate fuels will be investigated both analytically and experimentally.

In order to arrive at the optimum design of experimental equipment a computer model has been developed to be used in predicting the effect of geometric and experimental variables on heat transfer and profiles of pressure and temperature. Fuel is considered to flow through a cylindrical heat exchanger of constant cross-sectional area and increase in temperature prior to injection into the combustion chamber. The computer program predicts the pressure and temperature profiles of the fuel. Required input consist of pressure and temperature of the fuel at either end of the heat exchanger, mass flux of the fuel, heat flux profile at the wall tube dimensions, and fuel properties. The program is organized so that properties for any fuel can be inserted. The calculation is limited to single-phase flow; however, work is being done to extend the model to two-phase flow.

The model, one-dimensional along the tube axis, is based on a momentum balance, an energy balance and a mass balance:

$$v_z \frac{dv_z}{dz} + \frac{1}{\rho} \frac{dP}{dz} + \frac{2fv_z^2}{d} = 0$$
 (30)

$$v_z \frac{dv_z}{dz} + c_p \frac{dT}{dz} + \left(\frac{1}{\rho} + \frac{T}{\rho^2} \frac{\partial \rho}{\partial T}\right) \frac{dP}{dz} + \frac{4q}{Gd} = 0$$
 (31)

$$\rho v_z = G \tag{32}$$

where

z = axial distance from tube entrance

P = fluid pressure T = fluid temperature

d s tube diameter

vz = average velocity of fluid

p = fluid density

cp = fluid specific heat at constant pressure

G = average mass flux of fluid

f = friction factor

q = heat flux at the wall (positive value in the direction of increasing radius).

The friction factor for turbulent flow in a smooth pipe is given implicitly by ()

$$f^{-1/2} = 4.0 \log(\text{Re} \cdot f^{1/2}) - 4.0$$
 (33)

where Re = Reynclds number.

In the program, Equation (33) is solved by iteration to determine the friction factor for a known Reynolds number. The friction factor for laminar flow is

$$f = \frac{16}{3e} \tag{34}$$

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> Equations (30) to (32) were solved to yiell explicit expressions for the pressure and temperature derivatives in terms of the mass flux, the heat flux and properties of the fuel. Numerical integration of the derivatives then yields the pressure and temperature profiles. To obtain the derivatives the energy equation is simplified by subtracting Equation (30):

$$C_{\mathbf{p}} \frac{d\mathbf{T}}{dz} + \frac{\mathbf{T}}{\rho^2} \frac{\partial \rho}{\partial \mathbf{T}} \frac{d\mathbf{P}}{dz} + \frac{4q}{Gd} - \frac{2\mathbf{f} \mathbf{V}_z^2}{d} = 0$$
 (35)

Equations (30), (32), and (35) are now solved simultaneously. The fluid velocity in Equations (30) and (35) is replaced by the mass flux using Equation (32) and the resulting equations rearranged and made dimensionless:

$$\frac{1}{\rho}\frac{\partial\rho}{\partial P}\frac{dP}{ds} + \frac{d}{\rho}\frac{\partial\rho}{\partial I}\frac{dI}{dz} - \frac{\rho d}{G^2}\frac{dP}{dz} - 2f = 0$$
(36)

$$\frac{\rho^{2}c_{D}d}{G^{2}}\frac{dT}{dz} + \frac{Td}{G^{2}}\frac{\partial\rho}{\partial T}\frac{dP}{dz} + \frac{4\rho^{2}q}{G^{3}} - 2f = 0$$
 (37)

These equations are written in matrix form:

$$\begin{bmatrix} \mathbf{a}_{11} & \mathbf{a}_{12} \\ \mathbf{a}_{21} & \mathbf{a}_{22} \end{bmatrix} \begin{bmatrix} \mathbf{x}_1 \\ \mathbf{x}_2 \end{bmatrix} = \begin{bmatrix} \mathbf{b}_1 \\ \mathbf{b}_2 \end{bmatrix}$$
(38)

where

$$x_1 = \frac{\rho d}{G^2} \frac{dP}{dz} \tag{39}$$

$$x_{\mathbf{z}} = \frac{\rho^{\mathbf{z}} d}{C^{\mathbf{z}}} \frac{d\mathbf{T}}{d\mathbf{z}} \tag{40}$$

$$a_{11} = -1 + \frac{G^2}{\rho^2} \frac{\partial \rho}{\partial P} \tag{41}$$

$$\mathbf{a}_{12} = \frac{\mathbf{G}^2}{\mathbf{p}^3} \frac{\partial \mathbf{p}}{\partial \mathbf{T}} \tag{42}$$

$$a_{21} = \frac{T}{\rho} \frac{\partial \rho}{\partial T} \tag{43}$$

$$\mathbf{a}_{22} = \mathbf{c}_{\mathcal{D}} \tag{44}$$

$$b_1 = 2f \tag{45}$$

$$\mathbf{b_2} = 2\mathbf{f} - \frac{\mu \rho^2 q}{G^3} \tag{46}$$

The solution to Equation (38) is

$$x_1 = \frac{a_{22}b_1 - a_{12}b_2}{a_{11}a_{22} - a_{12}a_{21}} \tag{47}$$

$$x_2 = \frac{-a_{21}b_1 + a_{22}b_2}{a_{11}a_{22} - a_{12}a_{21}}.$$
 (48)

In the computer program Equations (47) and (48) are integrated numerically by the Runge-Kutts-Gill method. Fluid pressures and temperatures are then obtained as functions of the axial distance.

The critical velocity for choking is calculated at each integration step for comparison with the linear velocity. The critical velocity occurs as the pressure drop approaches infinty. Hence the critical velocity can be found by letting \mathbf{x}_1 approach infinity in Equation (47). The denominator on the right side is then zero and the critical velocity is given by rearrangement:

$$v_{c} = \frac{G_{c}}{\rho} = \left[\frac{\partial \rho}{\partial P} - \frac{T}{\rho^{2} c_{p}} \left(\frac{\partial \rho}{\partial T}\right)^{2}\right]^{-1/2}$$
(49)

where

v_c = critical velocity of fluid G_c = critical mass flux of fluid.

At periodic intervals during the integration a film heat transfer coefficient and a wall temperature are determined. The film coefficient is determined by any suitable heat transfer correlation, which can be inserted easily into the computer model.

In order to analyze data obtained in a resistance-heated heat exchanger, a pair of wall transfer coefficients, one for the heat generated and the other for the heat lost, are used to calculate the temperature drop across the wall. This temperature drop is given by

$$T_{1} - T_{0} = \frac{q_{C}}{h_{C}} + \frac{q_{L}}{h_{L}}$$
 (50)

where

$$h_{G} = -\frac{2k_{W}}{R_{1}} \left[\frac{1}{1 + \left(\frac{R_{1}}{R_{0}}\right)^{2}} + 2 \ln \left(\frac{R_{1}}{R_{0}}\right) \right]$$
 (51)

- heat transfer coefficient for heat generated by resistance heating in tube wall

$$h_{L} = \frac{k_{w}}{R_{1} \ln \left(\frac{R_{2}^{2}}{R_{0}}\right)}$$
 (52)

- heat transfer coefficient for heat transferred to surroundings

q₀ wheat generated by resistance heating expressed as an inside wall heat flux to the fluid

qL = heat lost to surroundings expressed as an inside wall heat flux from the fluid

Ti = inside well temperature

In - outside wall temperature

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Ri = inside tube radius

Ro = outside tube radius

k, - wall thermal conductivity

The inside wall temperature is determined by

$$T_{1} - T_{f} = \frac{q_{G} - q_{L}}{h_{f}}$$
 (53)

where

Tr = fluid temperature

hf - film heat transfer coefficient.

Comparison With Experiments

Experiments have been run at low and high heat fluxes. The calculations at low heat flux (1.56-1.63 x 10⁵ Btu/ft²-hr) compare favorably with experimental results. At high heat flux (4.50-8.22 x 10⁶ Btu/ft²-hr) the calculations are different from experimental observations. The predicted wall temperatures are higher than the observed values. Experimental results at low heat flux were obtained on a 0.277-in. diameter tube and at high flux on a tube of 0.0265-in. diameter.

Predicted temperature profiles at low heat flux are compared with experimental results in Figures 52 to 54. The mean bulk temperatures on the three figures lie in the subcritical, critical, and supercritical temperature regions. The calculated outlet bulk temperature in each figure agrees closely with the experimental value.

The profiles of the wall temperature were calculated from the heat flux, fluid temperature, and heat transfer coefficients based on different correlations. Two of the curves are based on the Dittus-Boelter correlation: 50)

$$Nu = 0.023 \text{ Re}^{0.8} \text{Pr}^{0.4}$$
 (54)

where

Nu = hrd = Nusselt number

 $Re = \frac{dO}{\mu} = Reynolds number$

 $Pr \cdot \frac{Cpu}{k} = Prandtl number$

k - fluid thermal conductivity

 μ = fluid viscosity.

Fluid properties for one curve are based on the mean bulk temperature and for the second curve on the average film temperature (the average of the mean bulk temperature and the inside wall temperature). The third curve is based on the Sieder-Tate correlation: 59)

$$Nu_{B} = 0.023 \operatorname{Re}_{B}^{0.49} \operatorname{Pr}_{B}^{1/3} \left(\frac{\mu_{B}}{\mu_{W}}\right)^{0.14}$$
 (55)

where the subscripts refer to properties at either the mean bulk temperature (B) or the inside wall temperature (W).

On each figure the measured wall temperatures on the top and bottom of the tube are close together at the tube inlet, diverge for 1 ft, and maintain a large difference for the remaining tube length. This temperature difference is probably due to secondary circulation inside the tube driven by density gradients. Hot fluid at the side rises to the top forcing colder fluid down across the centerline to the bottom of the tube.

The predicted wall temperatures generally tend to lie in the region of the bottom wall temperature. At subcritical bulk temperatures (T < 570.5°F, Figures 32 and 53) the Dittus-Boelter correlation based on the average film temperature shows less agreement with experiment than do the other two correlations. At ambient bulk temperatures the wall temperatures are high due to a large fluid viscosity, which decreases rapidly with increasing temperature. At the critical temperature, discontinuities in the fluid properties cause discontinuities in the heat transfer coefficient and hence the wall temperature. At supercritical temperatures (Figures 53 and 54) all three correlations predict essentially the same heat transfer coefficient. In Figure 54 the wall temperature for the Sieder-Tate correlation (not shown) lies between the wall temperatures predicted by the other two correlations.

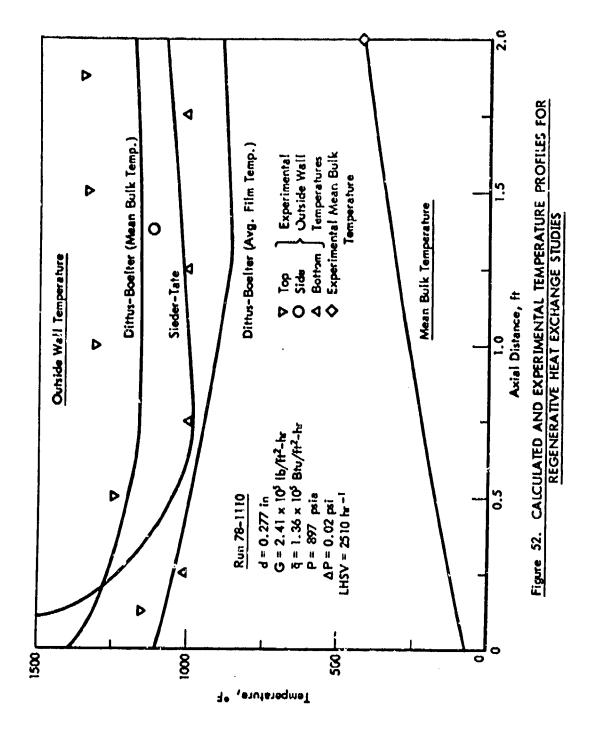
Predicted temperatures at high heat flux are shown in Figures 55 and 56. The mean bulk temperature is subcritical on both graphs. Outside wall temperatures based on three correlations are plotted for comparison with experimental temperatures. All three correlations predict conservative values of the heat transfer coefficient and hence high values for the wall temperature, although the temperatures based on the Dittus-Boelter correlation using fluid properties at the average film temperature are somewhat reasonable. The experimental wall temperatures are close together and show no evidence of secondary circulation. Actually the bottom wall temperatures are higher than the wall temperatures on top. This is probably due to a greater wall thickness at the bottom of the tube.

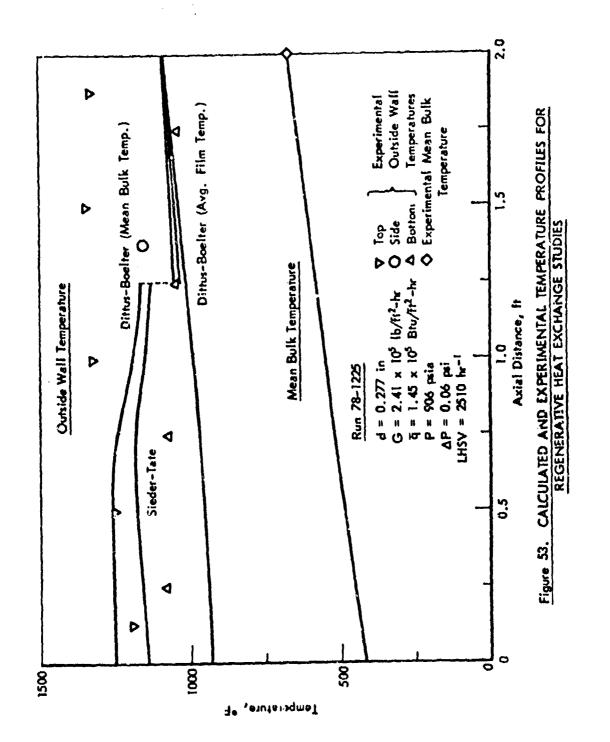
Shock Tube Studies of Ignition Delays of Hydrocarbons

The measurement of ignition delays has been extended to other high molecular weight hydrocarbons. Experimental methods and equipment have been described in previous reports.²⁾³⁾¹⁹ The latest measurements have been made on fuels that are possible candidates for regenerative ecoling of ramjet engines. The fuels used include both as-is components(DMD(dimethanolecalin), SHEILDYNE, and SHEILDYNE H) and mixtures (SHEILDYNE-Decalin and SHEILDYNE-Binor-S). Correlating equations were determined by linear regression for each fuel.

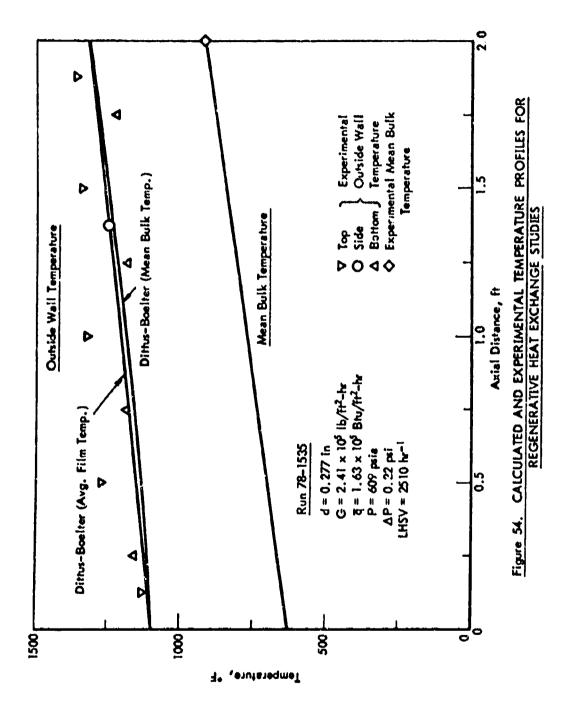
Experimental Work

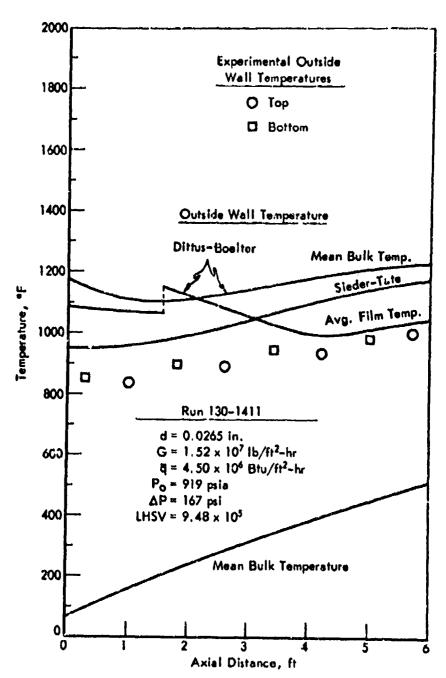
Mixtures were prepared at 80°C in order to have a sufficient concentration of fuel. The reaction section of the shock tube was heated to the same temperature, while the driver section was maintained at 45°C (except



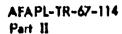


5 (2)





PROFILES FOR REGENERATIVE HEAT EXCHANGE STUDIES



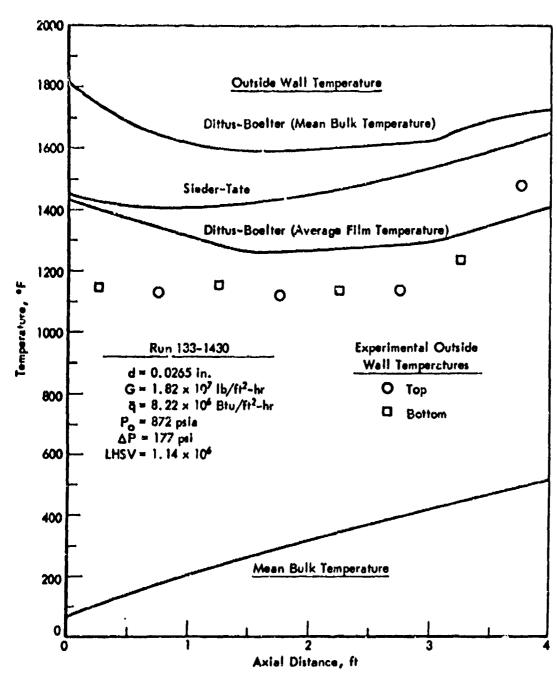


Figure 56. CALCULATED AND EXPERIMENTAL TEMPERATURE PROFILES
FOR REGENERATIVE HEAT EXCHANGE STUDIES

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near the diaphragm flange, where the temperature was also 80°C).

During each firing of the tube the apparent delay time of the mixture was determined by measuring the infrared emission from CO₂ at a frequency of 2350 cm⁻¹. The CO₂ emission was displayed as an oscilloscope trace, examples of which are the lower traces in the photographs of Figure 57. The upper trace in each photograph is the pressure history at the measuring station. Photographs (a) and (b) are characteristic of the results obtained on most of the shock tube runs. During the passage of the shock wave there is a sudden increase in the pressure, which is followed soon afterwards by a marked increase in CO₂ emission. A few of the runs with SHELLDYNE exhibited two breakpoints in the CO₂ traces, as shown in photographs (c) and (d). The initial increase was followed shortly by a dip and then a second increase in the CO₂ emission.

Data Analysis

The calculation procedure for analyzing data has been written as a computer program that furnishes the ignition delay and physical conditions for the combustion front. The calculation is based on the equations for conservation of mass, energy, and momentum in a one-dimensional shock wave. Perfect gas behavior and a logarithmic dependence of specific heat on temperature are assumed. The apparent delay time is corrected to the true delay time using the velocities of the shock wave and the following gas. Conditions at the combustion front are calculated from the conditions immediately behind the shock wave by assuming an exponential attenuation of the shock wave pressure. The pressure following the shock wave is assumed to decrease with distance according to

$$\frac{P_0}{P_1} - 1 = \left(\frac{P_{2, \text{ideal}}}{P_1} - 1\right) \exp\left(-\frac{Ax}{r_H}\right)$$
 (56)

where

P₁ = pressure ahead of shock wave

P2 = pressure following shock wave

x = axial distance

r_H = hydraulic radius of shock tube

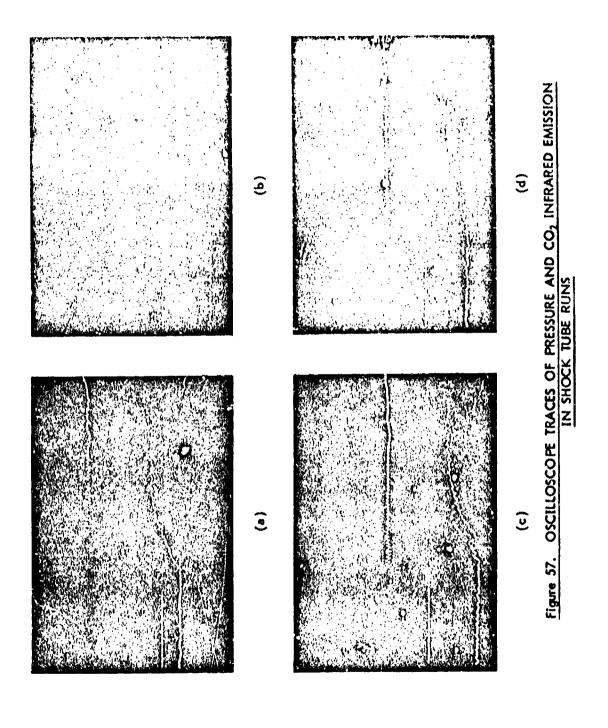
A = attenuation coefficient.

An attenuation coefficient of 0.001 was used, thich is in the range of 0.0005 to 0.001 estimated from attenuation coefficients for other shock tubes.

Results

Ignition delay times and conditions calculated from experimental data are listed in Tables 89 through 95 of the Appendix. The calculated delay times for each hydrocarbon or hydrocarbon mixture were fitted to correlating equations by multiple regression to determine the effects of independent variables. Temperature and oxygen concentration were found to be the only variables with significant effects. Hence the correlating equation used was

$$\ln r = b_0 + b_1 \ln c_{02} + \frac{E}{RT}$$
 (57)



where

t = ignition delay time, usec

co2 = oxygen concentration, g mole/liter

T = absolute temperature, "K

 $R = 1.987 \times 10^{-3} \text{ kcal/g mole-}^{\circ} \text{K}$

b1, b2 = correlating parameters

E = correlating parameter (activation energy), kcal/g mole.

Results of the correlation are listed in <u>Table 50</u>. The values of b₁ vary from -1.32 to -0.59 with most of the values close to -1. This led to the use of the following correlating equation:

$$\ln(\tau c_{O_2}) = b_O + \frac{E}{RT}$$
 (58)

Regression results for Equation (58) are listed in Table 59. Activation energies are just slightly different from those in Table 58. The standard error for $\ln(\tau c_{O_2})$ is only a fraction larger in Table 59 than in Table 58. Hence the simpler and more general Equation (58) is almost as good a fit as Equation (57).

Plots of ln(τco.) vs 1/T are shown among Figures 58 to 65 for various fuels. These illustrate the goodness of fit for Equation (58).

Figures 58 and 59 show the correlating equations for ignition of n-octane and Decalin. The delay times have been recalculated from earlier data³) using the computer program described above. There is some scatter of data on both plots. n-Octane seems to have no pattern for the scatter of its data. At low temperature, Decalin has delay times that correlate well. However at high temperature and low fuel-oxygen concentration there seems to be a dependence on the equivalence ratio or fuel concentration, since the delay time is almost proportional to the fuel concentration.

Results of shock tube runs for SHELLDYNE are plotted in Figure 60. In a few runs CO₂ emission increased at ignition and dipped afterwards (Figure 57, c and d). This was followed by another increase in the emission to a high level. The delay times for both of these increases fall along the two lines shown in Figure 60. Both lires are equations of the same form as Equation (58). Most of the points for SHELLDYNE fall along the upper line. However, the delay times for a few other runs and also the early delay times for those runs having two breakpoints in the CO₂ trace lie along the lower line. These runs with early ignition are indicated in Table 91 and are not restricted to a single mixture or set of conditions. The parameters for the two ignition delay lines are given in Table 59.

Figure 61 shows the ignition delays for a particular mixture of SHELLDYNE-oxygen-argon. The solid lines are the correlating equations for SHELLDYNE. Six CO₂ traces had two breakpoints each, three of whose delay times are the pairs of points connected by dotted lines in Figure 61. The other points were determined from CO₂ traces that had single breakpoints. The points on the graph appear to form two distinct lines similar to the ones shown. Hence it seems that the ignition of SHELLDYNE is complicated by two possible delay times. At the present time we have no satisfactory explanation

Table 58. IGNITION DELAY PARAMETERS IN EQUATION (57)

Fuel	Standard Error of ln(TCO2)	b _O	_b ₁	Standard Error of	E, kcal g mole	Standard Error of E
n-Octane	0,55	-17.19	-0.76	0.08	40.5	3.7
Decalin	0.61	-14.90	-0.59	0.07	39.2	3.9
SHELLDYNE	0.30	-22.09	-1.03	0.05	48.6	2.3
SHELLDYNE (Early Ignition)	0.26	-22.44	-1.05	0.10	46.4	3.1
SHELLDYNE H	0.35	-20.08	-0.93	0.05	43.9	1.3
DMD	0.62	-20.06	-0.95	0.19	45.6	4.2
SHELLDYNE- Decalin	0.60	-20.43	-1.10	0.13	40.7	4.0
SHELLDYNE- Binor-S	0.29	.27.77	-1.32	0.12	53.9	3.7

Table 59. IGNITION DELAY PARAMETERS IN EQUATION (58)

Fue1	Standard Error of In(1302)	_ъо_	E, kcal/g mole	Standard Error of E
n-Octan	0.56	-16.72	50.3	5
Decalin	0.67	-19.88	58.1	2.4
SHELLDYNE	0.30	-14.61	47.9	1.9
SHELLDYNE (Early Ignition)	0.25	-14.48	45.1	1.4
SHELLDYNE H	0.3;	-14.44	45.4	1.2
DMD	0.62	-13.84	46.2	3.3
SHELLDYNE- Decalin	0.60	-11.45	38.1	2.1
SHELLDYNE- Binor-S	٠٠37	-13.68	43.8	2.2

for this behavior of SHELLDYNE. However, it occurs with sufficient frequency to make it improbable that it is adventitious.

Ignition delay results for SHELLDYNE H and DMD are shown in Figures 62 and 63. SHELLDYNE H data correlate well with Equation (58), whereas the DAD has some scatter.

Additional shock tule runs at lower DMD-oxygen concentration would extend the data to higher temperatures and probably improve the correlation.

The correlating equations for the various fuels are compared in Figure 64. DMD and most of the SHELLDYNE points have similar ignition delay times. SHELLDYNE H has delay times that correspond to the early ignition delay times of SHELLDYNE. The delay times of n-octane and Decalin are shorter at high temperature. At lower temperatures these times are similar to those of SHELLDYNE H.

Shock tube runs were made for fuel mixtures of SHELLDYNE-Decalin and SHELLDYNE-Binor-S. Figures 65 and 66 show the results of these experiments. The least-squares linear fit is shown for each mixture along with ignition delay curves for other fuels. The SHELLDYNE-Decalin data at 1% fuel-oxygen concentration do not correlate well with the rest of the data. The data for the 1% fuel-oxygen concentration falls along the SHELLDYNE curve, while the rest of the data is more consistent with the delay times of Decalin and SHELLDYNE H. The SHELLDYNE-Decalin line is the best for all the mixture data. The SHELLDYNE-Binor-S data in Figure 66 correlate well. The best fit for the mixture is close to that of SHELLDYNE H.

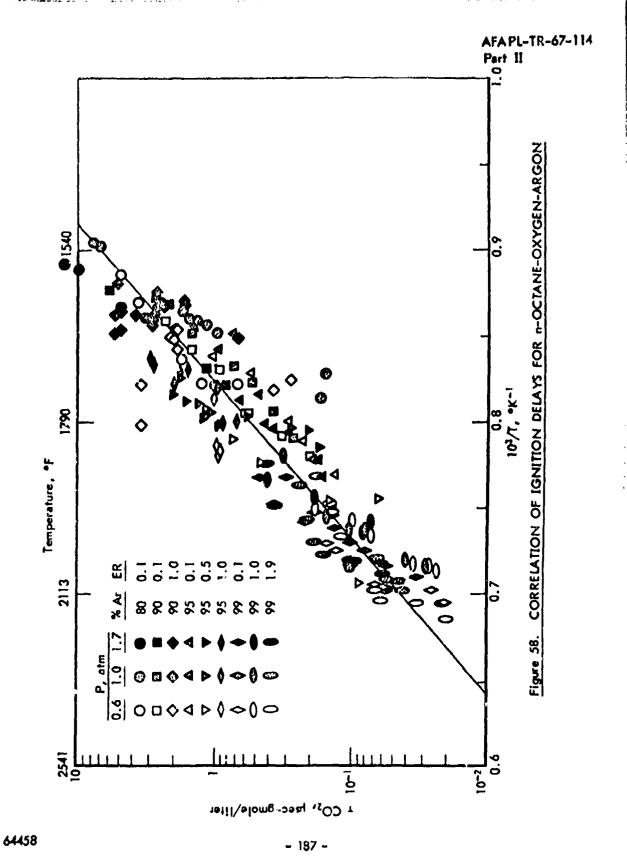
Estimation of Physical Properties of Hydrocarbons

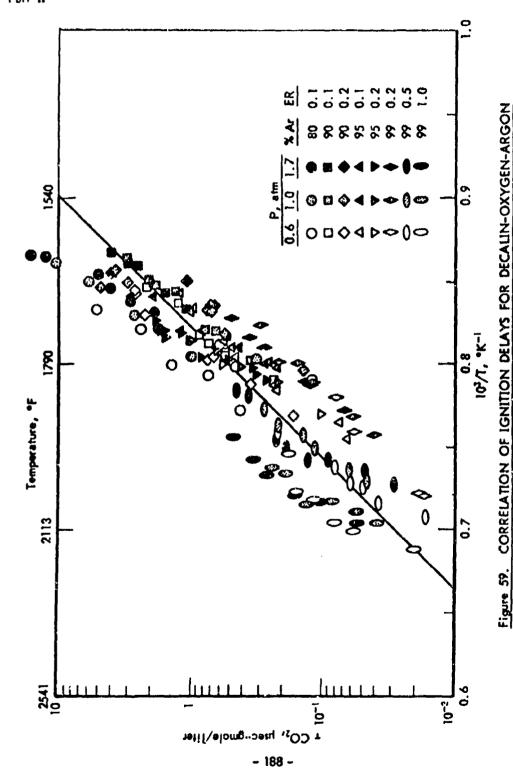
The available physical properties of a JP-7 type jet fuel (F-71), trans-Decalin, and SHELLDYNE are shown in Tables 96 through 104 of the Appendix. Most of the properties for F-71 and Decalin were estimated by the Sternling-Brown Properties Program. Most of the SHELLDYNE properties were calculated by a modification of the PPP-3 Physical Properties Program.

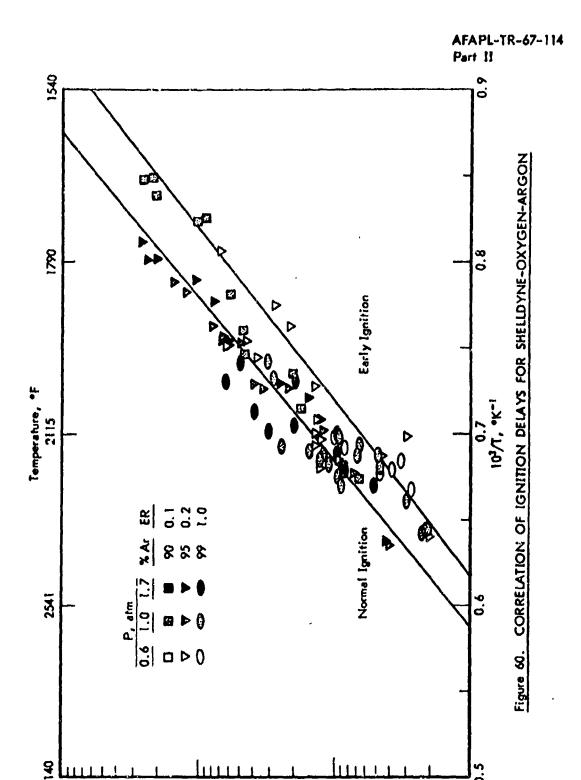
Critical properties of the normal paraffins were used in calculating properties of F-71, since previous analysis has shown the fuel to be composed mainly of paraffins with some naphthenes, olefins, and aromatics. In view of the possible variation in composition of available JP-7 type fuels, the assumption of a paraffinic composition should be sufficiently accurate for calculating properties, and the properties in Tables 96 through 98 should be representative of this fuel.

The current physical properties program, a modification of the A. I. Ch. E. program, is being revised, and when this is completed better estimates of the properties for trans-Decalin will be determined along with properties for cis-Decalin. In the meantime the properties in Tables 99 through 101 should be reasonably satisfactory for both isomers of Decalin.

Properties of F-71 and SHELLDYNE will also be recalculated with the revised program if they can be improved significantly. Otherwise the current properties should be adequate, since these two fuels are mixtures and the actual fuels available now or later may vary slightly in composition



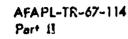


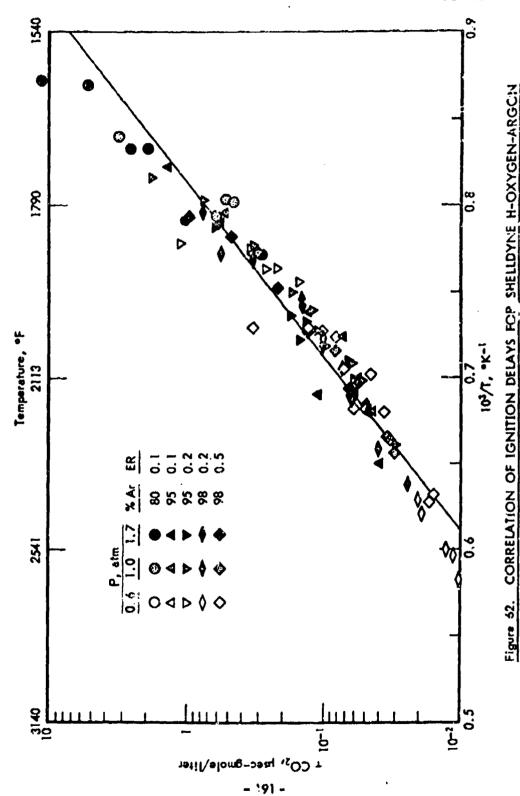


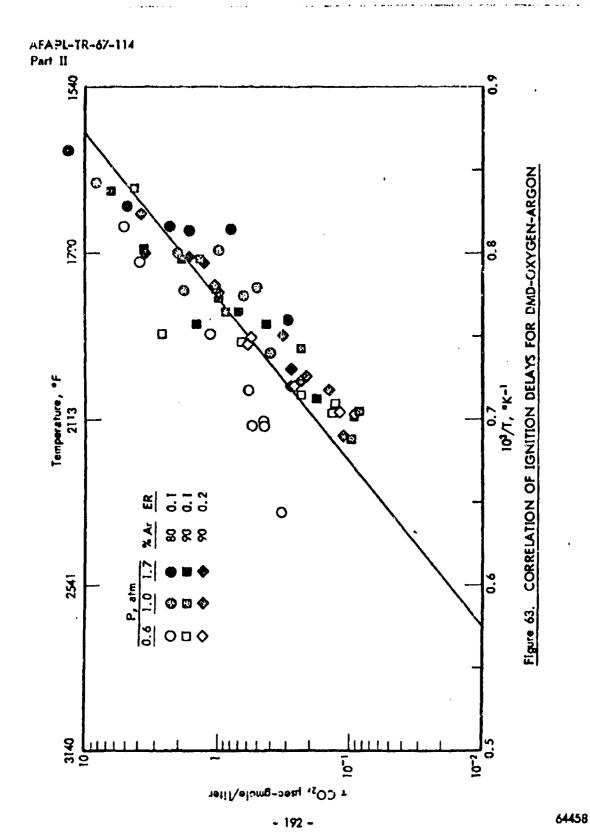
T CO2, prec-gnole/liter

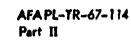
h CO3, prec-gnole/liter

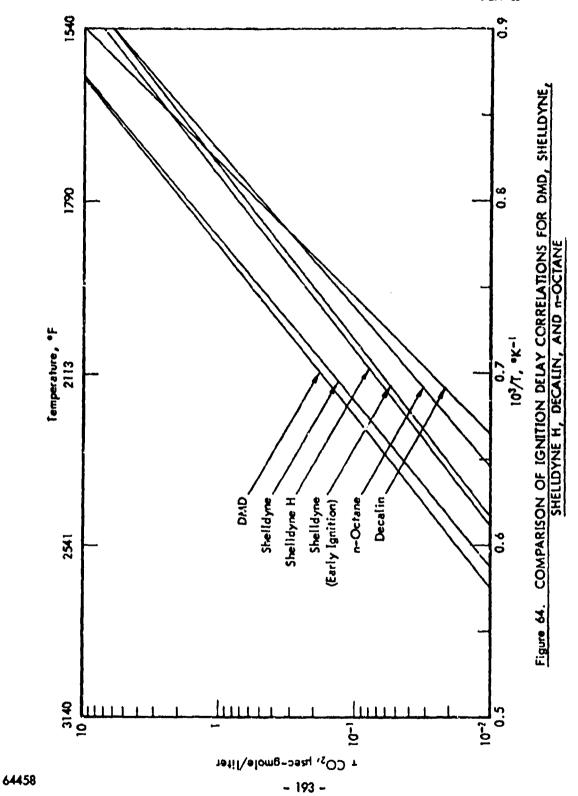
- 190 -

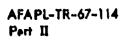


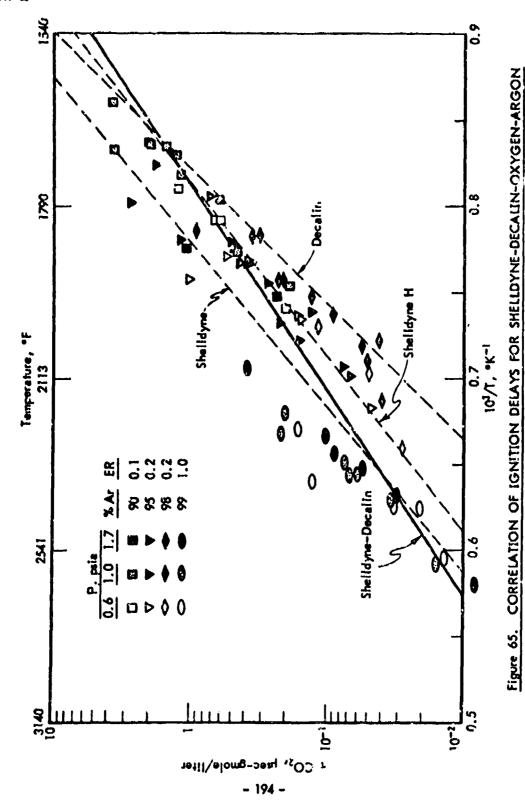


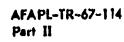


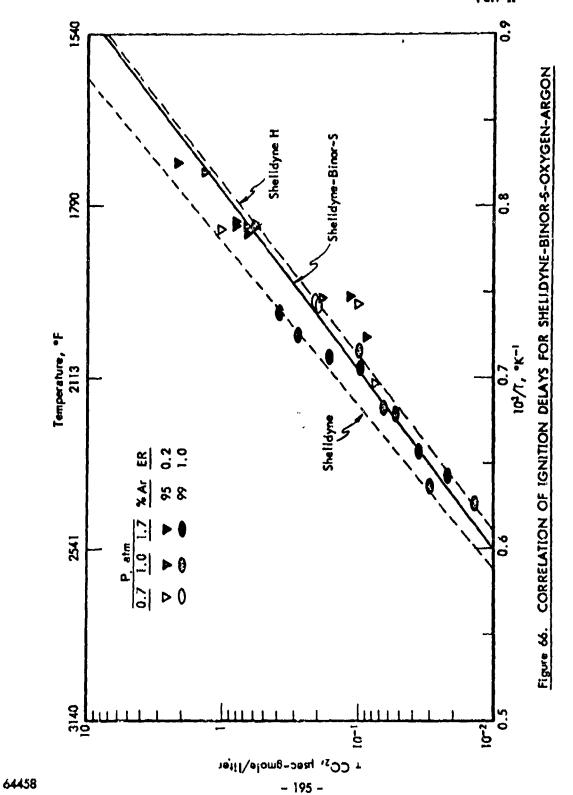












and properties.

The following section contains a summary of the various methods used for estimating physical properties. The list is essentially an identification of the formulas and correlations used in the calculation. The accuracy of these techniques is difficult to assess. The gas properties should be within 3% except in the critical region where errors of 10-30% may be likely. The liquid properties are less accurate: density 2%, heat capacity 10%, enthalpy 6%, heat of vaporization 5%, vapor pressure 3%, surface tension 10%, viscosity 10%, and thermal conductivity 15%.

Description of Estimation Methods

- 1. Compressibility factor calculated by the Ackerman modification of the Redlich-Kwong equation of state. 61)
 - 2. Liquid density from the Francis equation e2) for T ≤ T_c 60°F

$$\rho = A - BT - \frac{C}{E-T}$$
 (59)

and from the Guggenheim equation e3) for T > T_c - 60°F

$$\rho_{\mathbf{r}} = 1 + a(1-T_{\mathbf{r}})^{1/3} + b(1-T_{\mathbf{r}})$$
 (60)

- 3. Gas heat capacity at zero pressure from data or Rihani-Doraiswamy method. 64) Pressure effect from a modified form of the Redlich-Kwong equation of state.
- 4. Liquid heat capacity by numerical differentiation of liquid enthalpy.
- 5. Gas enthalpy at zero pressure from integration of ideal gas heat capacity. Effect of pressure from a modified form of the Redlich-Kwong equation of state.
- 6. Liquid enthalpy at saturation pressure by difference between gas enthalpy and heat of vaporization.
 - 7. Gas fugacity by numerical integration of

$$\ln \frac{f}{p} = \int_{0}^{P} \frac{Z-1}{P} dP \tag{61}$$

8. Gas free energy from the ideal gas free energy and the pressure effect on free energy

$$G - G^{\bullet} = RT \ln \frac{f}{f^{\bullet}}$$
 (62)

9. Cas entropy from the enthalpy and free energy at the same conditions

$$S = \frac{H - G}{T} \tag{65}$$

10. Gas specific heat ratio by differentiation of the Redlich-Kwong equation of state to evaluate

$$c^{D} - c^{\Lambda} = -\frac{\left(\frac{2L}{3\Lambda}\right)^{L}}{L\left(\frac{5L}{3\Lambda}\right)^{S}}$$
 (64)

11. Gas Joule-Thomson coefficient by differentiation of the modified Redlich-Kwong equation of state to evaluate

$$\mu_{\rm JT} = -\frac{1}{C_{\rm p}} \left(\frac{3H}{\delta P} \right)_{\rm T} \tag{65}$$

12. Gas sonic velocity from

$$V_{\mathbf{S}} = A \left[-\frac{\lambda}{\sqrt{\frac{9A}{9b}}} \right]_{\mathbf{r}/\mathbf{S}}$$
 (66)

using the Redlich-Kwong equation of state.

13. Heat of vaporization from the Theisen correlation 64)

$$\Delta H_{V} = a(T_{c} - T)^{n} \tag{67}$$

or the Watson correlation (n = 0.38).64)

14. Vapor pressure by the reduced Frost-Kalkwarf-Thodos correla-

15. Surface tension from an empirical correlation 55)

$$\sigma = a(1 - T_r)^b$$

or the MacLeod-Sugden correlation. 64)

16. <u>Gas viscosity</u> from the Chapman-Enskog theory with Kihara potential parameters. Gas Correction for pressure by the Stiel and Thodos method. 65)

17. Liquid viscosity from an ASTM standard viscosity-temperature chart for $T < \frac{200 \, ^{\circ} F_{s}}{100 \, ^{\circ} F_{s}}$

$$\ln \mu = a + \frac{b}{T}$$
(68)

for 200°F < T < 0.7 T_c, and from the Stiel and Thodos dense gas method for T_r > 0.7.84)

- 18. Gas thermal conductivity from the Chapman-Enskog theory. 64) Correction for pressure by the Stiel and Thodos method. 65)
- 19. Liquid thermal conductivity from the Robbins-Kingrea correlation for $T_r < 0.9$ and from the Stiel and Thodos dense gas correlation for $T_r > 0.9$.

Present Status and Future Projections

- 1. A considerable effort has been made during this year to gather data to allow us to advance Decalin to the same status as a candidate fuel as is now occupied by MCH. Decalin is a promising candidate fuel in its own right and would be valuable as a component of a mixed fuel in which the Decalin is incorporated to decrease the vapor pressure or modify the freeze point or viscosity. The Decalin system is considerably more involved than is MCH because of complexity of the dehydrogenation reactions. We have apparently been successful in obtaining kinetic data in bench-scale apparatus under "isothermal" conditions (vide infra) for Decalin over Pt/Al₂O₃. This will enable us to complete the mathematical model and then run Decalin in the FSSTR in order to check it.
- 2. Preliminary examination of the thermal reactivity of the high density fuel SHELLDYNE in our all metal bench-scale reactor equipment revealed that it displays susceptibility to cracking reactions even at as low a temperature as 800°F; the extent of reaction depending on the contact time in the reaction zone. Although the reaction is relatively clean at low conversion, at high conversions (i.e., high temperature or long reaction time) coking can occur. The reactivity of the SHELLDYNE can be greatly reduced by hydrogen treatment, which is readily accomplished. Thus, under similar conditions, the ratio of SHELLDYNE to SHELLDYNE H reaction is around 1500 while the reactivity of SHELLDYNE H is comparable with that of Decalin. Since the effect of hydrogen treating on the heat of combustion and physical properties of SHELLDYNE is relatively minor, it is evident that SHELLDYNE H should be used in any application involving cooling. While this conclusion must be tentative and will await further work in the mini-FSSTR under more representative conditions, it is unlikely that it will be modified. There is some indication that the metal used in our bench-scale equipment had c catalytic effect on the decomposition of the SHELLDYNE. The hydrotreating also improved the thermal stability of SHELLDYNE as measured in the SD/M-7 coker, being superior to Decalin in tube deposits and somewhat inferior in pressure drop effects. The next step will be to determine the behavior of SHELLDYNE H in the mini-FSSTR in order to get basic data for heat transfer calculations.
- 3. Other areas of interest in connection with the development of endothermic fuels involve studying the rates and extent of dehydrogenation of DMD, SHELLDYNE H, BCH (bicycloheptane), hydrogenated fulvenes, adamantane and higher naphthenes over our standard catalyst. It will also be of interest to study dehydrocyclative reactions for molecules involving spatially favored hydrogen atoms. An attempt will also be made to bring about rapid dehydrogenation of TIB in the presence of a volatile strong acid which could serve as a vapor phase catalyst.

4. Although a substantial number of the 536 catalysts that have been evaluated under our catalyst development program have shown greater activity (and in some cases greater stability) than the standard Pt/Al₂O₃ catalyst, we have not achieved the sought-for order of magnitude increase in activity desired nor the hoped-for cheap equivalent catalyst. Recent work has been concerned with the production of catalysts containing two or more catalytic elements to which one or more auxiliary elements have been added in an effort to promote substantially higher catalytic activity. Few synergistic effects have been noted as a result of these attempts. We intend in the future to systematically broaden our experimentation by increasing still further the number of elements included in a single catalyst.

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A few catalysts which had been shown to be more active than the standard catalysts in MICTR tests were further examined in the bench-scale apparatus under a broader range of conditions with results substantially in agreement with the screening test results. Further testing of this type will be done as more improved catalysts become available.

slong two routes. One, to develop a catalyst which can be coated on the inside of a heat exchanger tube. The other, to provide a catalyst precursor which can be dissolved or dispersed in the fuel and will be converted in the heated zone into a volatile or dispersed catalyst having the requisite activity to bring about the heat sink reaction. A number of thixotropic formulations have been devised which, when dried and platinized, result in a catalyst with at least as much activity as our standard catalyst and at the same time can be converted into a slip which can be conclided to the inside of a catalyst tube and fixed by calcining to give an adherent coating. The coating is then platinized in situ. Experiments to check the catalytic activity of such surface catalysts have yielded some encouraging preliminary information as to the practicability of this approach. The best catalysts will be examined further in FSSTR experiments.

In the area of possible dispersed, soluble or vaporizable catalysts some success has been achieved in the past in static experiments carried out in a heated autoclave. These and additional possible catalysts which have been prepared or purchased will be further checked in two different types of apparatii which are now in hand, namely a heated injection autoclave and a pulse type reactor. Preliminary experiments in the pulse reactor have turned up three model compounds which had some activity. It is expected that a large number of materials will be examined during the next year.

6. The mathematical model devised to represent the catalytic dehydrogenation of MCH based on an axisymmetric packed reactor successfully represents the reaction under various flow and reaction conditions (although it had to be modified to accept the 2-ft reactor data). We have been for some months past attempting to develop a similar model for the Decalin system utilizing kinetic data obtained in the bench-scale reactor with diluted beds to supply the basic kinetic parameters. So far it has not been possible to successfully represent the reaction over a wide enough range of reaction variables. Accordingly, some additional data was obtained in the bench-scale reactor using a differential reaction system in order to reduce the uncertainty introduced by the indeterminate temperature variations. The model now seems to be rounding into shape. Similarly, we are attempting to develop a

satisfactory model for a regenerative heat exchanger operating under the conditions anticipated for the near term supersonic combustion missile application. Applying the model to the data obtained in the mini-FSSTR results in satisfactory representation of the data in the supercritical region but is less successful in the critical and subcritical regions. Additional work on this model will be done as more experimental data are obtained.

- In order to provide heat flux conditions closer to those that might be encountered under application conditions, a two-foot by three_sighths inch OD tube was constructed for the FSSTR and operated at heat fluxes up to 600,000 Btu/hr/sq ft, using R-8 catalyst with MCH feed. By introducing liquid MCH at 70°F into the reactor tube. it was possible to operate at close to the maximum heat load of the section; however, it was evident that catalyst deactivation was beginning to occur at temperatures as low as 900°F. This was accompanied by a rise in the exit fluid temperature as well as the outside wall temperature and by a decline in the MCH conversion. Final fluid temperatures of 1250°F were encountered and a maximum wall temperature of 1500°F. For the system involved, it appears that rapid coke buildup should be anticipated in any region where tube wall temperatures are greater than 1350°F and fluid temperatures above 1150°F are found. These results emphasize the necessity for developing more thermally stable and more active catalysts. Fortunately, these have been provided by our catalyst development program and will be tested in future work in the high flux section.
- 8. Heat transfer, pressure drop, and coking data are being obtained for candidate missile fuels using a short small diameter electrically heated section substituted for the down stream section of the FSSTR (this has been dubbed the mini-FSSTR). The application being modeled here is that of a nonreactive heat sink in which the fuel will not exceed 900°F at the outlet with an inlet pressure not exceeding 1000 psi. During the recent year tubes 4 or 6 inches long by 26.5 mils ID have been used for the study section and nitrogen, MCH and water have been used as test fluids. With each test section fairly high heat fluxes and flow rates have been achieved. Each of the first three tubes used failed for one reason or another, the first by plugging with carbon and the next two by burnout, although not at the most severe condition encountered. It is suspected that fluctuations in the flow rate due to uneven exit valve operation or to surging in the feed pressure may have been the cause of the failures. It has been shown that the radial variation of wall temperatures which were of alarming proportions in the case of earlier work with a 3/8 inch OD tube section also exists in the small diameter tubes, although of a lesser magnitude. In the latter case, however, we have found that this variation is due to unequal thickness of the tube wall and, hence, unequal generation of heat by passage of the electrical current. Whether this was also the case with the 3/8 inch tube remains to be seen. Additional identical 4 or 6 inch by 26.5 mil sections have been manufactured. These were used for experimentation with water and MCH; Decelin and SHELLDYNE H will be the next test fuels, followed by F-71 and methane. Following this some experimentation with larger tube size sections will be done. A maximum heat flux of 8.5 x 10° Btu/hr/sq ft has been achieved to date.
- 9 We have two devices that we have been chiefly relying on for determining the thermal stability of fuels in this study: the SD coker, and the CAFSTR. The former is essentially an improved ASTM coker but we have operated it in a recycle mode in order to conserve fuel. In the past, the

feed section has been at atmospheric pressure and a Zenith pump has been used as a combination metering and pressure developing device. Because of the considerable difficulty we have had with this pump and the amount of pump wear which has occurred, we modified the SD coker to operate with the whole system under test pressure, using the pump merely as a metering device. This modification seems to be quite successful and we have used it for obtaining data on a number of fuels, including SHELLDYNE, SHELLDYNE H, and Decalin, as noted above.

No operational problems have been encountered with the CAFSTR. Runs which have been made to date on it have shown that the design concepts were quite sound. The problem here is to rate the tubes in a meaningful manner. The three possible types of evaluations favored are deposit removal by solvent action, deposit removal by combustion and deposit thickness evaluation by electron recoil. All of these are, hopefully, nondestructive to the tube. A large number of solvents were evaluated for their efficacy as deposit removers at 100°C. The only significantly promising solvent found was dimethyl formamide. This will be checked against other evaluation methods. Although the conditions necessary for utilizing combustion as an evaluation method were checked out previously utilizing the CAFSTR tube heater as the heat source for the combustion, we are also evaluating a method utilizing an external furnace as the source of the combustion, with the expectation that this will be less destructive to the tube, and, particularly, to the tube heater. Other devices, including a H2 ring burner, a plasma torch and a laser are under consideration.

10. In the past we have been limited in our ability to determine the ignition delay of candidate fuels in our shock tube on the basis of fuel volatility, since we require a vapor phase mixture of the fuel, oxygen, and argon to charge into the tube. In order to extend our capabilities into the higher molecular woight materials such as SHELLDYNE and dimethanodecalin (DMD) we modified the tube to utilize electric heaters to increase the ambient temperature of the fuel, oxygen, diluent mixture prior to carrying out the shocking experiment. It was also necessary to eliminate all cool crannies which might provide sites for condensation. Thus, it was necessary to replace the precision pressure gauge used in making up the mixture with a pressure transducer. We have now operated the tube at an ambient temperature of 80°C to compare the ignition delay of DMD, SHELLDYNE and SHELLDYNE H. We find this to be the order of decreasing ignition delay (with the SHELLDYNE H having a delay approximating that of n-octane). A presently inexplicable peculiar behavior was noted on a number of occasion; with SHELLDYNE, which gave two correlatable ignition delay times in the same experiment. The shorter delay was approximately the same as that exhibited by SHELLD'NE H while the longer delay was about the same as that shown by DMD. Future work is intended to go to higher ambient temperatures (and, hence, higher equivalence ratios) for these and other high molecular weight molecular types, and to higher pressures for a range of molecular weights. We also hope to improve the sensitivity of the IR detection system to enable us to follow more precisely the production of CO2 and hence the combustion rate during the reaction.

11. We are keeping in constant touch with improvements being made in the various correlation schemes for computer production of properties of various molecules. We are utilizing two proprietary programs, the Sternling

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Brown and the PCP program as well as the A. I. Ch. E. program (in part). We have also taken advantage of a proprietary effort to calculate the properties of molecules from the molecular structure. We utilized this in connection with the correlative programs to determine the properties of SHELLDYNE over a broad range of temperature and pressure. We have also similarly calculated the properties of a JP-7 type fuel (F-71) and trans-Decalin. Unfortunately, each time the properties are calculated, we have to make the proviso that these are subject to revision at some future date if better experimental data or improved calculation methods become available. We must do so in the present instance.

12. Decalin has a number of advantages as an endothermic fuel (compared to MCH), mainly in its lower vapor pressure, higher density, and its better thermal stability and lubricity. In order to have an adequate supply of this material on hand, we obtained thirty drums from the Air Force fuel bank (RAF-161-60). This had been in storage since 1960. In spite of the fact that it was inhibited, care taken in the selection of the material and the low temperature storage conditions, its color and thermal stability had deteriorated substantially. Laboratory work showed that it could be regenerated by silica gel treatment, so this operation was carried out on the thirty drums, resulting in a material of practically pristine quality. Some of this material has been supplied to Pratt and Whitney and to the Air Force at Wright Field. Interestingly, the impurity removed seemed to be fairly simple in character and relatively volatile containing three major components of about the same emergence time from a GLC column as naphthauene. A considerable quantity of this material was described from the silica gel column used in the thirty drum purification and will be examined Jurther to try and identify the materials responsible for the poor thermal stability behavior of the Decalin. The material also had vesicant properties, causing a severe dermetitis to susceptible individuals.

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Immoription of the Pulse Reactor

The pulse reactor was a 1/4 in. OD stainless steel tute (no. 30h) 9-1/4 in. long and 0.028 in. vall thickness. Swagelok Tees were fastened at each end and one arm of the Tee served as an injection port. A rubber septum (GLC type) was held in place by the fitting nut and the feed was injected through this septum from a syrings. A five inch length of the reactor tube was surrounded by a secondary furnace liner and the whole was heated by an electric furnace. The secondary liner had seven radial drilled holes for thermocouples, and the holes were located as shown in Figure 67. A schematic diagram of the pulse reactor is shown in Figure 68.

All lines were 1/4 in. OD stainless steel tubing (no. 304). About 28 in. of line just prior to the reactor was wrapped with heating tape and constituted a grs preheater. About 8 in. of the preheater section was filled with quartz chips (10-20 mesh size).

In the pulse reactor system the carrier gas was motered through a rotameter (Figure 68) and passed through the preheater section and into the reactor. The exit gas passed into a manifold and then into the GLC. The purpose of the manifold was to maintain the exit gas pressure slightly greater than the gas pressure in the GLC. This was done by adjusting the pressure control valve and the vent valve. The manifold was wrapped with heating tape and was maintained at 302° to 356°F. The injection port temperature was about 450°F. The pressure control and the vent valves were needle valves (Hoke No. 1315) and the GLC valve was a lever operated valve (Hoke No. 490).

To carry out an experiment the reactor was brought to temperature and the carrier gus flow rate, reactor pressure and manifold pressure were adjusted by means of the appropriate flow control valves. Then with inert gas flowing to the SLC a pulse was injected through the lower injection port and subsequently analysed. This gave an analysis of the starting material. A pulse was then injected in the 'op injection port, passed over the catalyst and analysed.

In this system the space velocity was obtained from the inert gas flow rate. Figure 69 shows the pulse reactor system with the secondary furnace liner in place; Figure 70 shows the GIC analysis system.

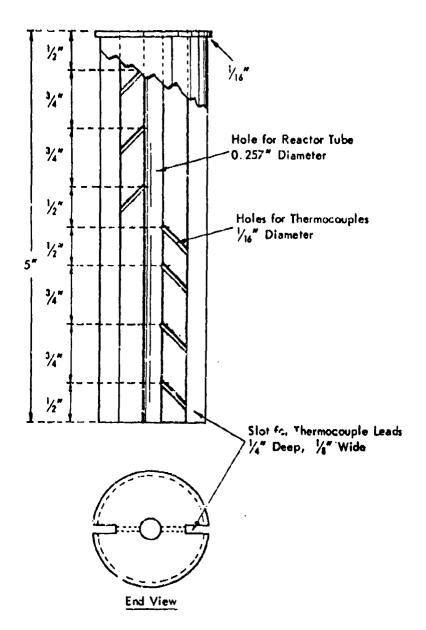


Figure 67. SECONDARY FURNACE LINER FOR PULSE REACTOR

William Strategy

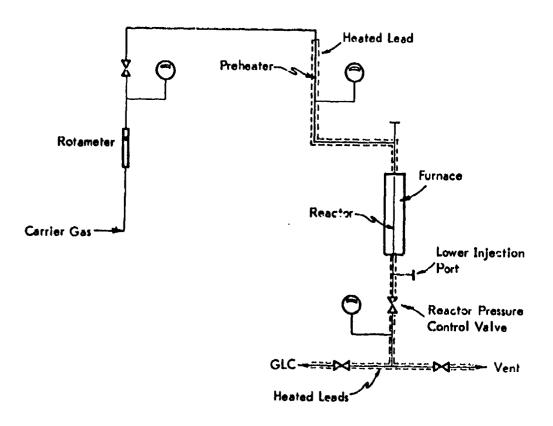


Figure 68. PULSE REACTOR: SCHEMATIC

AFAPL-TR-67-114 Part II

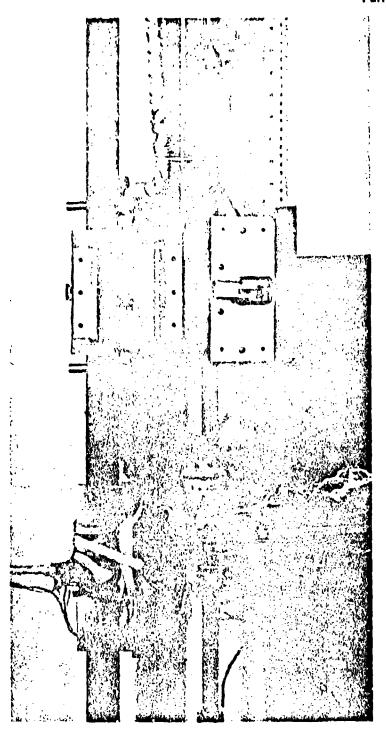
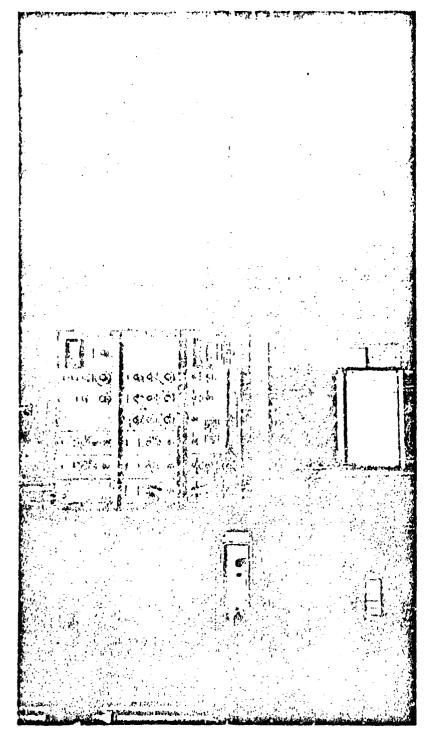


Figure 69. PULSE REACTOR SYSTEM



e 70. GLC ANALYSIS SYSTEM

化成场形式,这些工作工具

Table 69. DEHYDROGENATION OF DECALIN OVER VARIOUS CATALYSTS

F-113 Decalin 7 ml 19 atm 100 30 min Feed: Catalyst Volume: Pressure: LHSV: Resction Period:

		ł																
Cctalyst	14 71 4010 (Standary	16 Pt on Burster 0106 Alueina tanderé Catalyst)	ther lyst)		uor -rB		14	American Cyanadide American Pillal; 0,85 Pt	ij	Sinc.	Sinclair-Daker AD-150; 0,6% Pt	H &:		30260-16			12250-45	
Am 16, 1732.	120	121	27	Ж	প্ত	82	*	ñ	×	r.	я	2	×	×	15	X	2	2
Temperatume, 'f	2,5	3	į									_		_			}]
ofile	657.66	18 18 18 18 18 18 18 18 18 18 18 18 18 1	\$ 20 X		_												X 2 7 3	25.5 25.5 25.5
	6.00 6.00 6.00 6.00 6.00 6.00 6.00 6.00	8 6 6 6 6 6 6 6 6 6 6 6 6 6 6 6 6 6 6 6	77°	2 - 62) 12-119 12-119	65145 69145 79145	\$ 8 t	10 10 10 10 10 10 10 10 10 10 10 10 10 1	\$ 8,8 \$ 8,8 \$ 81	55 55 55	25.23.2 8 8 4 4	69772 69772 708-67	7 N.7	943	334 355 555	12.5 13.5 13.5 13.5 13.5 13.5 13.5 13.5 13	865 634 634	£23	1. 1.28 1.28
Many "F, cetalyst bed	٥	ĸ	ថ	*	5	કૂ	9	8	19	-	4.	Ħ	6	Ħ	6	•	~	ជ
Product Analysis, \$4 trans-DilN		23.3	, , , ,	\$	7.52	24.7	5.7	z.s	3.3	27.4	23.4	13.8	33.2	8	23.1	35.5	9,12	8
35 - 1 (a)		80.0	200	7.0	29.5	21.5	8.0	23.7	8	9.4	90	20.0	8	21.7	6:11	12.	19.4	5.3
Ĕ.		9.9	-	16.6	15.5	1,5	15.8	, ë	8.6	13.0	10.8	6.10	0	13.9	ğ	3.60	0.00	20.1
Mephthelen	2.5	3.2	20.0	19.4	0 Q	7.0	50.5	7.6	9 9	3 6	0.85	o &	25.2	1. 5.	ئ ئ		3	9 5
Crecief, 15quid	100	0 0	400	w 0	30	400		500	# # 0 0	200	N 0	000	0.0	00	000	1	33	200
rield rift, &	6.1	6.2	.; .;	16.2	12.7	3	15.4	2.2	6,2	9.6	1.0.4 4.0.4	?	17.6	14.5	2.01	3.5	14.5	17.71
Selectivity for 8, %	75.0	0.48	8	53.8	69.5	79.7	57.2	73.6	78.7	8.8	2.5	78.4	59.0	72.5	d. J.	9	63.6	9.00
DEN CONMERSION (3.8	79.1	15.3	χ.,	6.54	1.1.	XÝ.	P. B.	84	8	7.60	8	ė,	1.64	e. e.	9	13	979
First Order Rate Constant, sec 1	0.38	8,0	69.0	0.45	09.0	ŗ.	2,43	8,0	5.73	, A	?	9.	0.50	69.3	36.0	% 6	0.74	0.97
E, act, heal/wole	-1.6		1	8.7	~	١	-3		•	-;i	٠.	·	10.		•	_ of	-56	•

Table 61. DEHYDROGENATION OF DECALIN OVER SHELL:
46 LARCHATORY CATALYST

F-113 Decalin 74.6% cis-DHN 25.0% trans-DHN 0.4% THN Catalyst Volume: 7 ml Feed: THSV: 100 Reaction Time: 30 min

Pressure: 10 atm

kun Ho.	4	~ ~~~~	-1054A-9		
Temperature, Y	``				
Block	842	932	1022	1112	1202
Yel i	725-29	784-90	853-60	941-52	1094-98
Catalyst Bed Profile	604-17	639-53	573-89	716-40	601-66
-	597-95	630-35	662-66	704-07	774-81
	606-10	640-44	676-80	725-25	
	5:7-21	655-57	698-59	750-50	837-30
ATmax* Ta)	13	14	16	24	65
Product Analysis, Su	()				
trens-life!	34.2	29.7	24.5	18.6	11.9
cis-DIN	23.7	20.1	16.2	13.4	11.3
ր ե)	0.0	0.0	0.0	0.1	0.9
THE	20.5	16.4	11.1	6.1	5.0
ლხ)	0.0	0.0	0.0	0.0	0.0
N	21.4	33.6	47.9	60 . S	67.6
Մ Ե.)	0.2	0.2	0.3	0.4	0.5
Cracked, Ilq.	0.0	0.0	0.0	0.5	2.2
Yield THN, 🏍	20.1	16.0	10.7	5.7	4.6
DOM Conversion, Zu	41.9	50.0	59.2	67.9	81.2
Selectivity for THM - N, Fu	99.5	99.2	99.0	98.1	89.0
Rate Constanta					
Zero Order, eta, sec ⁻¹	4.02	5.05	6.30	7.73	10.22
First Order, sec 3	0.52	0.70	0.95	1.29	2.10
E _{ect} , kcsi/sole	 7	' ₋₈			

a) Maximum increase in catalyst but temperature during the 30 minute run.

b) Unidentified.

Table 62. DEHYDROGENATION OF DECALIN OVER SHELL, 108
LABORATORY CATALYST

F-115 Decalin 74.6% cis-DHN 25.0% trans-DHN C.4% THN Catalyst Volume: 7 ml LHSV: 100 Feed: Reaction Time: 30 min

Run No.: 10548-5 Pressure: 10 atm

Temperature, 'F					
Block	842	932	1022	1112	1202
Wall	709-11	768-72			1008-22
Catalyst Bed Profile	617-28	653-66	689-705	732-54	822-946
•	615-21	651-55			806-37
	624-26	662-66			830-33
}	635~37	676-80	725	784-80	862-56
Δr _{max} , •Fa)	11	13	16	22	124
l max.					
Product Analysis, %	(i				
trans-DHN	30.5	27.4	23.3	18.1	11.6
cis-DHN	32.1	27.8	22.4	19.1	16.9
Up)	0.0	0.0	0.0	0.0	0.5
THN	14.6	10.5	7.0	4.9	5.2
Մ Ե)	0.1	0.1	0.1	0.1	0.3
N ₁	22.6	34.0	46.8	56.8	62.0
ΰ δ)	0.1	0.2	0.3	0.5	0.4
Cracked, liq.	0.0	0.0	0.1	0.5	3.1
Yield THN, Tu	14.2	10.1	6.6	4.3	4.8
DHN Conversion, Aw	37.0	44.6	54.1	62.4	71.1
Selectivity for THN + N, fw	99.5	99.3	98.7	97.9	94.0
Rate Constants					
Zero Order, stm, sec 1	3.53	4.43	5.69	6.91	8.42
First Order, sec 1	0.44	0.59	0.82	1.08	1.48
Eact, kcal/mole	جــــــــــــــــــــــــــــــــــــ	.1>			

a) Maximum increase in catalyst bed temperature during the 30 minute run.

b) Unidentified.

THE ASSESSMENT OF THE PARTY OF

Table 63. DEHYDROGENATION OF DECALIN OVER SHELL 107A LABORATORY CATALYST

Catalyst Vol.: 7 ml LHSV: 100

Feed:

F-113 Decalin 74.6% cis-DHN 25.0% trans-DHN

Reaction Time: 30 min Run No.:

10342-196

0.4% THN

			Press	ure: 10) e cm		
Run No.	196	197-1	198-1	198-2	199-1	199-2	199-3
Tesperature, T				}			
# lock	842	932	1022	1112	1112c)	1112c)	11120)
Vali	714-25	788-801	866-96	945-1078	1085-87	1087	1087-92
Catalyst Bud Profile	646-62	687-712	740-815	801-1064	1071-58	1058-54	1054-72
	612-17	642-55	680-707	722-1036	1045-54	1054	1054-67
	610-14	642-48	676-86	722-959	986-1031	1031-40	1040-60
	525-28	662-68	705-09	761-907	923-1006	1005-35	1035-53
ATmax, "Fa)	16	25	75	314	-	-	•
Product Airelysts, Su	Ì						
trans-DHN	30.8	27.5	23.2	19.2	22.3	24.4	25,2
c1#-DHN	30.0	25.2	22.5	26.5	52.2	59.3	60.9
(φ)	0.0	0.0	0.3	3.9	9,5	7.0	6-2
THE	17.2	13.6	9.i	6.3	3.7	2.3	2.1
լլե)	0.0	0.0	0.1	0.4	2.2	1.9	1.5
H asks	21.8	32.5	44.5	42.3	7.7	2.7	1.5
ub)	0.2	0.2	0-2	0-2	0.0	0.0	0.0
Cracked, IIq.	0.0	0.0	8-1	1.2	2.4	2.4	2.6
Yleid THM, Su	16.8	13.2	8.7	5.9	3.3	1.9	1.7
OHM Conversion, Sw	36.9	45.1	54.1	\$4.1	25.2	16.0	13.5
Solectivity for THM - N, Su	99.5	99,6	98.7	69.4	43.9	28.9	23.8
Rute Constants							
Zero Order, eta, sec ^{*1}	3.71	4.68	5.86	5.44	-	. į	•
First Order, soc"1	0.47	0.63	0,85	0.93	-]	- [•
E _{act} , kcml/mole	 6,	5					

a) Maximum increase in catalyst bed temperature during the 30 minute run-

b) Unidontified.

c) Rune 199-1, 199-2, 199-3 were consecutive rune of 10-sinute duration following Run 198-2.

Table 64. DEHYDROGENATION OF DECALIN OVER SHELL 21A LABORATORY CATALYST

Catalyst Vol.: 7 ml Feed: F-115 Decalin
LHSV: 100 74.6% cis-DHN
Reaction Time: 30 min 25.0% trans-DHN
Run No.: 10342-180 0.4% THN

Pressure: 10 atm LHSV: 100

Temperature, *F Block Wall Catalyst Bed Profile	842 705-09 637-50 617-15 621-17 624-21	932 770-77 680-702 640-51 644-53 653-57	730-66 676- 89	1112 918-25 794-869 729-47 736-43 752-48	1200 1010-82 916-1121 806-968 804-51 820-31
Δī _{maχ} , • _F e.)	13	22	36	50	205
Product Analysis, for trans-DHN cis-DHN ub) THN ub) N Ub) Cracked, liq.	31.0 29.8 0.0 18.7 0.1 20.3 0.1	26.1 27.6 0.0 14.3 0.3 31.5 0.2 0.0	21.4 25.5 0.0 10.3 0.3 44.2 0.2	16.8 21.4 0.4 6.4 0.0 54.4 0.3	11.5 18.0 1.6 5.1 0.0 60.4 0.3 3.1
Yield THN, fw	18.3	13.9	9.9	6.0	4.7
DEN Conversion, Au	39.0	46.1	54.9	61.6	68.8
Selectivity for THN + N, fw	99.5	98.1	98.7	98.3	95.0
Rate Constants Zero Order, atm, sec 1 First Order, sec 1	3.68 0.47	4.60 0.61	5.80 0.88	6.88 1.07	8.38 1.42
Eact, kcal/mole	<6.	<u> </u>			

Maximum increase in catalyst bed temperature during the 50 minute run.

b) Unidentified.

Table 65. DEHYDROGENATION OF DECALIN OVER SHELL 105B LABORATORY CATALYST

Feed:

Catalyst Vol.: 7 ml LHSV: 100 F-115 Decalin

Reaction Time: 30 min Run No.: 188 74.6% cis-DHN 25.0% trans-DHN 0.4% THN

Pressure: 10 atm LHSV: 100

	,				
Temperature, 'F		!			
Blook	842	932	1022	1112	120?
Wall	707-09	763-65	827-33	898-910	1028-1148
Catalyst Bed Profile	626-33	658-69	700-20	756-831	974-1150
	621-26		<i>6</i> 93-98		869-1106
	624-26	,	693-98	740-52	842-1064
	632-35	665-69	707-09	759-63	851-1006
ΔT _{max} , · _F a)	7	11	20	75	257
	}				
Product Analysis, fw		ac -			
trans-DiN	32.4	26.9	21.0	15.9	12.9
oie—CHN ub)	28.1	25.2	22.2	21.7	51.9
THN	0.0	0.0	0.0	0.0	6.5
np)	17.0	13.6	9.5	5.4	5.4
N .	22.4	34.1	0.0 46.9	0.0	0.1
np)	0.1	0.2	0.3	56.3 0.3	45.3
Cracked, liq.	0.0	0.0	0.1	0.4	3.7
Tield Tin, fo	16.6	13.2	9.1	5.0	5.0
DHN Conversion, #w	39.3	47.7	56.6	61.9	55.0
Selectivity for THN + N, fw	99.5	99.4	99.2	99.2	91.7
Rate Constants		1			
Zero Order, atm, med 1	3.71	4.73	5.91	6.83	-
First Order, sec 1	0.47	0.64	0.87	1.07	
Empt, koal/mole	<8.8	>			

Maximum increase in catalyst bed temperature during the 30 minute run.

b) Unidentified.

GIC Analysis System

Analyses for napithene dehydrogenation systems have been made almost entirely with an F & M GLC (Models 710 and 5754A) using a packed column and a thermal conductivity detector. This technique is useful for rapid analyses and for determining trace amounts of material; but for analyzing mixtures of close boiling compounds, such as cis and trans isomers of poly methyl and ethyl substituted cyclohexanes, a capillary column is needed. As future work on fuel evaluation will involve analyses of multicomponent systems having a multiplicity of close boiling components, it was expedient to convert our analysis system to the capillary technique.

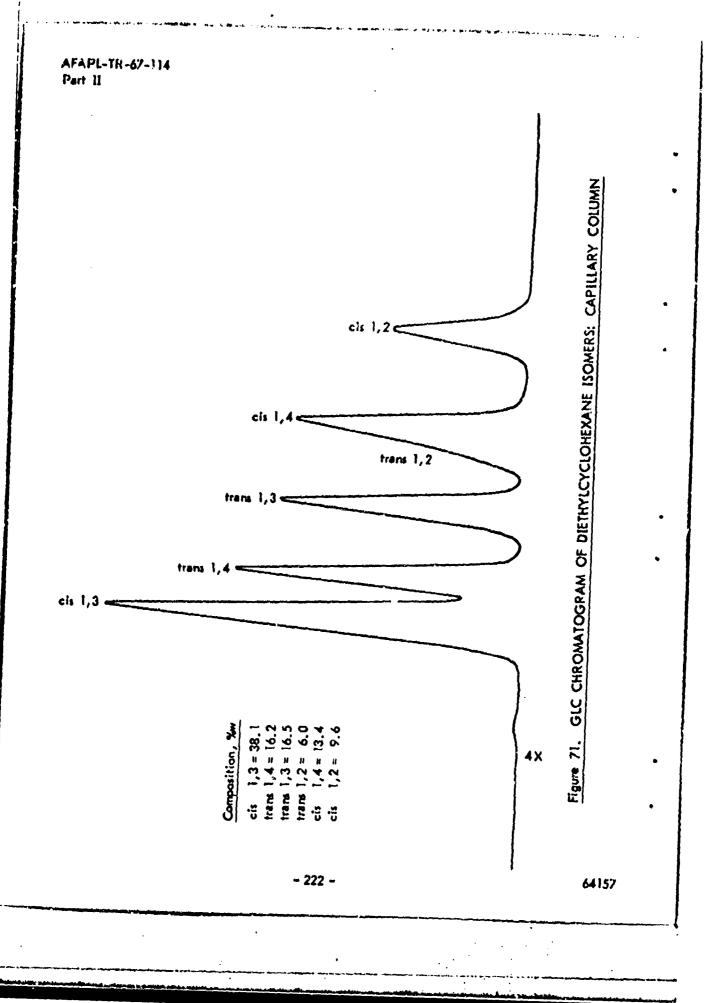
At present our analyses are being made with an F & M GLC, Model 5754A using a hydrogen flame detector and a 165° stainless steel capillary column, 0.010° diameter, coated with 20% phenyl ethers in DC 710 silicone. The superior resolution of the capillary column over a packed column is shown by chromatograms of a mixture of diethylcyclohexane isomers and their cis and trans species that were analyzed by the capillary (Figure 71) and packed column (Figure 72) techniques.

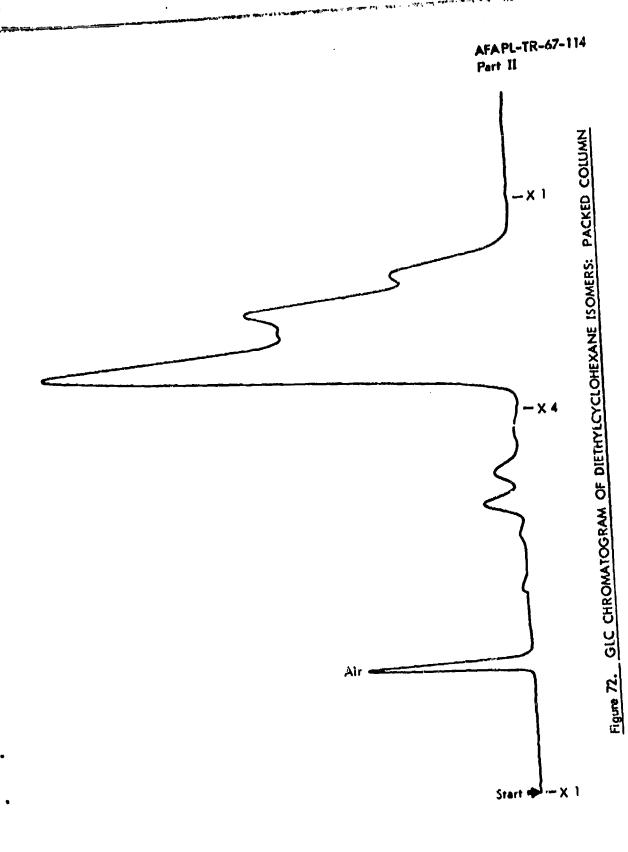
Micro Catalyst Test Reactor Data

The micro catalyst test reactor (MICTR) and the operational techniques used for screening candidate catalysts have been described in the Appendix of the last Annual Report. Catalysts are tested with MCH at LHSV 150 and 662,752 and 842°F and with n-heptane at LHSV 10 and 842 and 932°F, in all cases at 10 atm pressure without added hydrogen. Figures 87 through 89 of ref 19 show the apparatus in detail, except that the feed line pressure gauge has been eliminated to reduce feed line hold up and a check-valve installed to prevent accidental "blow-back".

The original Wheelco integrating recorder (for GLC peaks) became unserviceable and was replaced after run 291 by a Honeywell integrating recorder. This in turn was replaced by a Westronic integrating recorder starting with run 531. Starting with run 277, a small screen was placed above the bottom slotted separator in the MICTR tube. This was done to prevent an occasional small catalyst or quartz particle from plugging the slot which would cause an increase in back-pressure and a corresponding decrease in conversion over catalysts under test. At least once a week the reference catalysts 9874-24 or 159 were retested and the results used as a base point for performance evaluation of candidate catalysts tested for that particular week. The test results are shown chronologically in Tables 65, 67, 68 and 70. Table 69 gives preliminary results with coating candidates as described earlier. A few runs have been left out where mechanical or instrumental problems occurred and the validity of the data are in doubt. In such cases the catalysts were retested.

a) An excellent discussion of the usefulness of various GLC techniques is given by L. S. Ettre in "Open Tubular Columns in Gas Chromatography", Plenerr Press, New York, 1965.





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Table 66 MCH DEHTUROGENATI'N WITH VARIOUS CATALYSTS IN MICTR: RUNS 263-450

Ŷ

The state of the s

June-August 1967.	10 Atm pressure; estalysts reduced in H2	for 20 minutes et '196'F; CLC samples norm-	
Period:	Conditions:		

aliy taken at 3-, 8- and 13-minute operation at each block temperature.

0.9 ml catalyst diluted with 1.1 ml quarts chips; IMSY 100 (catalyst and quartz particles 10.20 mesh unless otherwise noted). Catalyst volume:

E E	Cat. Jugat IL	Catalyst		¥ MCB	f MCH Conversion, *P	E, °F
છું		Description	¥t, 8	299	752	248
(5)	10260-27	52 Metal/R-8 Al 30.		6	_	0
492	10280-271	Bimetallic, 5.25 metals/R-8 A120.		, 00 00 00 00 00 00 00 00 00 00 00 00 00	12,11,16	44.24.40
8	287.28	12 Pt/R-8 Al 20 (ref)	CH C	8 28		3 1 0
% 	2574-7	1% Pt/support type No. 1	;	1 % 1 %		86. 35. 30 10. 10. 10. 10. 10. 10. 10. 10. 10. 10.
%	2-1195	Š	0.77	31.32.31	65.60.56	8 8 8
8	7-+166	type	0.76	0.76 36.33.30	65.65.59	87,84,82
202	10280-278	Simetallic, 5,2%2-8 41203	,	13.11.6	19.12.10	20, 20, 25,
בעז	9874-7	1% Pt/support type My. 1	7,0	30.32.37		84 00 BA
272	10280-27C	(R-8 412	74.0			3,26,56
2734)	9874-199B	4% Metal/support type No. 10)d)	0,38	× × ×		74 74 74
27.12	9874-161B	type No.	8	0.28 17.18.19		66.60
27.70)	1 9871-1618 ⁰)	type	0.42	× 5. 18		25 17 67
2784	1028c-24c	type type	8, 1	8 X X X X X X X X X X X X X X X X X X X		(5,14,01 (5,64,61)
27947	9874-161B		8	808		8 9
8	9874-132	(Simil	5 0	1 % C % C % C % C % C % C % C % C % C %		12,20,22
€	62-03701	15 P./R-3 Alz0s (errored to (Oz gas)	1 5	38		862
83	9874-948	4% Metal/support type No. 2	3 2 5	8,8 8,8 8,8		9,69
83	10280-244	.g	7,0	10, 10, 10, 10, 10, 10, 10, 10, 10, 10,		88 85 87
₹ ~	10280-24A	4C-01) C -04	200	7 4 4		
8	1508-80E	بر چ	2 5	0.6		(0, (1, 52
2867	9874-24	15 Pt/R-8 11 - (Tage)	2.0	8,5		5,6,50
₹	10280-45	5% Metal/support type No. 6 (repeat of	, c	2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2	C. C. C.	3,8
		•	;	20132103		Citalic
3	LHSV 200.					1000

(Continued) Bottom of catalyst hed soreemed from rum 276 om to prevent plugging of slotted bottom spacer. Metal tetramina hydroxide carbomated before impregnation. Muffled at 1082'F in air. क्टिइ

Table 66 (Contd-2). MCH DEHITROGENATION WITH VARIOUS CATALYSTS IN MICHR: RUNS 263-450

F		Catalyst		EX 12	Conversion,	9. ° 6.
£	CEURLYST NO.	Description	¥. 8	5652	752	548
32.		45 Metal/support type No. 2 (as rea'd) 45 Metal/support type No. 2 (as rea'd)	0.41	16,18,15	16,39,34 56,61,53	67,60,55 85,80,75
350	25 1-1-7-1-7-1-1-1-1-1-1-1-1-1-1-1-1-1-1-1	*t/R-8 AlgOs *etal/support type No. 1*)		8,8,8 33,32,30	46,48,45 62,58,54	72,55,53 87,82,78
72.00 2 m b)		fetal/82% support type No. 18% support type No.		28,31,32	29,31,31	37,33,31
100		/82% support 18% support	1.1	6, 1, -	7, -, -	17,48, -
32k 323b)	9874-948 12280-658	type No.	0.36	28,28,27 38,43,42	51,45,48	89,76,71
324	10280-658	\$2.50 \$7.50 \$7.50		22,24,21	45,42,39	64,62,59
325 326b)	9874-140 £ 10280-684	support type No. port type No. ? support type No.	0.37	27,86,82 36,86,33	50,43,40	71,59,57
327		type No.		17		
38	10280-49 9874-140A	2% Metal/support type No. 1 10% Metal/support type No. 1		26, 26, 39 65, 36, 30	54, E4, C6	73,79,74
33007		80 Mesh, 90% Pt-10% ir screen, Pt black		- 000	0.0	
332	10280-504	Binetallia, 4, 34/R-8 A1203		5,86,55	54,53,49	68,53,39
334	45-4126 9874-24	15 Pt/R-8 Algos (ref) 16 Pt/R-8 Algos	0.42	28,89,27 35,35,35	8. 8. 7. 7.	71,71,85
335	10280-718			8,8,8	36,37,36	75,72,75
336	10290-71D	5 5 5 5 5 5	0.34	33,21,32	56,52,53	69,69,70
337 338	10280-661 10280-75E		0.F	8,8,8	29,26,26	0,0,0
		41% support type No. 1, 18% binder type Ho. 6	;	7767607	06,00,00	8) 60,69
a) Cat	Catalytic coating IESV 50.	ating on exterior of 8" long, 0.11" diam hypodermic tube.	le tube.			(Continued)

b) LESV 50.
c) Catalyset diluted to 4 ml instead of usual 2 ml volume.

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3 253-430 Table 66 (Contd-4). HOR DEHYTHOURANTION WITH WARIOUS CATALIERS IN MIGHE: -

September-November, 1967
10 atm pressure; catalysts reduced in hydrogen at 796°F, CLC samples normally taken at 3-, 8-, and 13- minute operation at each temperature. Volume 0.9 rl catalyst diluted with 1.1 ml quartz chips. IHST = 100 Conditions: Catalyst: Perfod:

catalysts and quarts 10-20 mesh unless otherwise noted).

Me. Me. Description Tt, g 662 752 842 353 10280-776 25 metal/805 type I support - 20f type 6 binder 0.43 0 <th>Ş</th> <th>Catalyst</th> <th>Catelyat</th> <th></th> <th></th> <th>\$ ECH Conversion,</th> <th>in ere</th> <th>'s, "F</th> <th></th>	Ş	Catalyst	Catelyat			\$ ECH Conversion,	in ere	's, "F	
10280-776 25.5 metal/805 type support - 207 type & binder 0.75 25, 25, 24 60, 7, 58 89, 10280-777 3.55 metal/type & support 0.59 0.43 0.99 0 0 0 0 0 0 0 0 0	ė	Ko.	Description	7t, g	299	151		278	
1/280-775 3.5% matel/type 6 support 0.43 0.43 0 0 0 0 0 0 0 0 0	35;	13280-718	25 meta: (80% type I support - 20/ type 6 binder	0.76	26,	ន	82	88, 85, 82	Γ
10280-770 4.6 metal/type è support (raf.) 1931-24 15 Ft/R-8 Ala-0, support (raf.) 1931-25 15 15 15 15 15 15 15 15 15 15 15 15 15	352	14280-77C	3.5% metal/type 6 support	0.43	0				
9874-24 If Pt/R-8 Alg-support (ref.) 9874-24 If Pt/R-8 Alg-support (ref.) 9877-24 If Pt/R-8 Alg-support (ref.) 9877-24 If Pt/R-8 Alg-support (ref.) 9874-7 If Pt/Ol04 Alg-support (ref.) 9874-7 If Pt/R-8 Alg-support (ref.) 9874-7 If If If Rt-8 Alg-support (ref.) 9874-7 If	323	2	4.4 metal/type e support	0.39	0	6		6	
9874-24 [[PtfR-B Al_04 support (ref.)] 9874-24 [[PtfR-B Al_04 support (ref.)] 9874-7 [[PtfOl04 Al_04, support (ref.)] 9874-7 [[PtfOl04 Al_04, support (ref.)] 9874-7 [[PtfOl05 Al_04, support] 9874-7 [[PtfOl05 Al_04, support] 6646-37 [Simstailie, 32.8, 50.2] [no support) 6646-37 [Simstailie, 32.8, 50.2] [no support] 6646-38 [Simstailie, 32.8, 50.2] [no support] 6646-37 [Simstailie, 32.8, 50.2] [no support] 6646-38 [Simstailie, 32.8, 50.2] [no support] 6646-39 [Simstailie, 32.8, 50.2] [no support] 6646-14 [Simstailie, 32.8, 50.2] [no support] 6646-15 [Simstailie, 32.8, 50.2] [no support] 6646-16 [Simstailie, 32.8, 50.2] [no support] 6646-10 [Simstailie, 1, 15/R-8 Al_04, support] 6647-16 [Simstailie, 1, 15/R-8 Al_04, support] 6648-10 [Simstailie, 1, 15/R-8 Al_04, support] 6649-10 [Simstailie, 1, 15/R-8 Al_04, support] 6640-10 [Simstailie, 1, 15/R-8 Al_04, support] 6	324		If Pt/R-8 Alada support (ref.)	0.21	≅.	3,		55, 53, St	
9874-7 17 PP (0104 Algos) 5603-181 Pinetaille, 38.6, 31.6 (no support) 6646-31 Binetaille, 39.8, 30.2 (no support) 6646-31 Binetaille, 70.4, 29.6 (no support) 6646-64 Binetaille, 70.4, 29.6 (no support) 6646-14 Pinetaille, 52.4, 47.6 (no support) 6646-14 Pinetaille, 32.9, 62, 0.17 (no support) 6646-13 Frientaille, 32.9, 62, 0.17 (no support) 6646-14 Pinetaille, 22.9, 62, 0.17 (no support) 6646-10 Pinetaille, 22.9, 62, 0.17 (no support) 6646-10 Pinetaille, 25.2, 73.8 (no support) 6646-10 Pinetaille, 52.7, 46.37 (no support) 6646-10 Pinetaille, 52.7, 46.37 (no support) 6646-10 Pinetaille, 17 PPR-8 Algos 10280-790 Binetaille, 1, 15/R-8 Algos 102	355	5874-24	15 Pt/R.B Algds support (ref.)	0.0	2	\$		77. 74. 63	
9874-7 if Pt/OlG4 Ai_0. 5603-181 Simetailie, 38.6, 31.6 (ne support) 6646-37 Simetailie, 38.6, 31.6 (ne support) 6646-37 Simetailie, 38.6, 31.6 (ne support) 6646-48 Simetailie, 32.2 21.6, (no support) 6646-48 Simetailie, 32.4, 47.6 (no support) 6646-64 Simetailie, 32.9, 62, 0.1 (no support) 6646-14 Simetailie, 32.9, 62, 0.1 (no support) 6646-16 Simetailie, 32.9, 62, 0.1 (no support) 6646-17 Simetailie, 32.9, 62, 0.1 (no support) 6646-18 Simetailie, 32.9, 62, 0.1 (no support) 6646-19 Simetailie, 32.7, 46.3 (no support) 6649-17 Simetailie, 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	356	2674- 7	I'E Pt/Olot Algo,	0.7	R	23		84, 76, 74	
\$603-181 Simetailie, 98.6, 31.6 (ne support) \$646-31 Simetailie, 79.8, 50.2 (no support) \$646-31 Simetailie, 70.4, 29.6 (no support) \$646-64 Simetailie, 70.4, 29.6 (no support) \$646-64 Simetailie, 70.4, 29.6 (no support) \$646-114 Simetailie, 32.9, 62, 0.15 (no support) \$646-114 Simetailie, 32.9, 62, 0.15 (no support) \$646-114 Simetailie, 32.9, 62, 0.15 (no support) \$616-1173 Trimetailie, 32.9, 62, 0.15 (no support) \$616-1173 Trimetailie, 32.9, 62, 0.15 (no support) \$616-1173 Trimetailie, 22.2, 73.85 (no support) \$616-1173 Trimetailie, 25.2, 73.85 (no support) \$616-1173 Trimetailie, 25.2, 73.85 (no support) \$616-1173 Trimetailie, 23.7, 46.35 (no support) \$616-117 Simetailie, 23.7, 46.35 (no support) \$616-117 Simetailie, 11 15/R-8 Algo, \$610-117 Simetailie, 11 15/R-8	357	9874- 7	IF Pt/010s Alzoz	(q:)*0	ដ	ង		75. 67. 66	
E76-119 Bimetallie, 39.8, 50.27 (no support) 6646-54 Bimetallie, 70.4, 29.67 (no support) 6646-64 Bimetallie, 70.4, 29.67 (no support) 6646-114 Bimetallie, 70.4, 29.67 (no support) 6646-114 Bimetallie, 52.4, 47.67 (no support) 6187-173 Trimetallie, 32.9, 62, 0.17 (no support) 6187-173 Trimetallie, 32.9, 62, 0.17 (no support) 6187-173 Trimetallie, 22.2, 73.87 (no support) 6299 6740-16 Bimetallie, 26.2, 73.87 (no support) 6740-16 Bimetallie, 53.7, 46.37 (no support) 6740-16 Bimetallie, 53.7, 46.37 (no support) 6740-16 Bimetallie, 53.7, 46.37 (no support) 6740-173 If 74/R-8 Alg. Alg. Alg. Control) 6740-174 If 74/R-8 Alg. Alg. Control) 6740-175 Bimetallie, I If/R-8 Alg. Control) 6740-175 Bimetallie, I If/R-8 Alg. Control) 6741 I4, 12, 12 31, 23, 22 47, 10, 28, 28, 29, 48 16, 20, 10, 280-796 Bimetallie, I If/R-8 Alg. Control) 6742 IS 13, 23, 22 47, 10, 20, 20, 22, 23, 23, 24, 20, 20, 28, 22, 24, 20, 28, 20, 28, 23, 29, 20, 20, 20, 20, 20, 20, 20, 20, 20, 20	38	\$603-181	Simetaille, 58.6, 31.6 (no support)	23.0		•		0	
6646-84 Bimetallic, 78.2, 21.8, (no support) 6646-64 Bimetallic, 70.4, 29.65 (no support) 6646-14 Bimetallic, 52.4, 47.65 (no support) 6646-114 Bimetallic, 52.4, 47.65 (no support) 6187-1738 Trimetallic, 52.4, 47.65 (no support) 6187-1738 Trimetallic, 22.9, 62, 0.15 (no support) 6187-1738 Trimetallic, 22.2, 73.85 (no support) 6646-104 Bimetallic, 26.2, 73.85 (no support) 6646-104 Bimetallic, 26.2, 73.85 (no support) 6646-104 Bimetallic, 53.7, 46.35 (no support) 6749-161 Bimetallic, 53.7, 46.35 (no support) 6640-758 Bimetallic, 1, 15/R-8 Algo, 10280-752 Bimetallic, 1, 15/R-8 Algo, 10280-752 Bimetallic, 1, 15/R-8 Algo, 10280-755 Bimetallic, 1, 15/R-8 Algo, 10280-756 Bimetallic, 1, 15/R-8 Algo, 10280-757 Bimetallic, 1, 15/R-8 Algo, 10280-756 Bimetallic, 1, 15/R-8 Algo, 10280-756 Bimetallic, 1, 15/R-8 Algo, 10280-757 Bimetallic, 1, 15/R-8 Algo, 10280-756 Bimetallic, 1, 15/R-8 Algo, 10280-757 Bimetallic, 1, 15/R-8 Algo, 10280-756 Bimetallic, 1, 15/R-8 Algo, 10280-757 Bimetallic, 1, 15/R-8 Algo, 10280-756 Bimetallic, 1, 15/R-8 Algo, 10280-757 Bimetallic, 1, 15/R-8 Algo, 10280-756 Bimetallic, 1, 15/R-8 Algo, 10280-757 Bimetallic, 1, 15/R-8 Algo, 10280-757 Bimetallic, 1, 15/R-8 Algo, 10280-758 Bimetallic, 1, 15/R-8 Algo, 10280-759 Bimet	329	£1.46-119		3.28	9	0		0	
6646-64 Simetalile, 70.4, 29.6% (no support) 6646-14 Simetalile, 32.4, 47.6% (no support) 6646-114 Simetalile, 52.4, 47.6% (no support) 6187-1738 Trimetalile, 32.9, 62, 0.1% (no support) 6187-1738 Trimetalile, 32.9, 62, 0.1% (no support) 6187-1738 Trimetalile, 25.2, 73.8% (no support) 6646-104 Simetalile, 25.2, 73.8% (no support) 6749-161 Simetalile, 53.7, 46.3% (no support) 6749-161 Simetalile, 53.7, 46.3% (no support) 6749-161 Simetalile, 53.7, 46.3% (no support) 6749-161 Simetalile, 11 [\$\frac{1}{2}\text{Al}	ğ	6646-37		0.75	0	-		0	
6645-46 99.4% metal (no support) 6645-114 Simetallie, 52.4, 47.6% (no support) 6187-1738 Trimetallie, 32.9, 62, 0.1% (no support) 6187-1738 Trimetallie, 32.9, 62, 0.1% (no support) 9874-24 [\$\tilde{x}^2 \tilde{x}^2 \ti	38	19-9199		0.15	0	-		· •	
6146-114 Statistic, 52.4, 47.6% (no support) 6187-1738 Trimetallic, 32.9, 62, 0.1% (no support) 6187-1738 Trimetallic, 32.9, 62, 0.1% (no support) 9874-24 [\$\tilde{5}.7\tilde{4}.\tilde{8}.\tilde{8}.\tilde{6}.6	352	81-5193	(no support)	38°	6	-		0	
6187-1738 Trimetallie, 32.9, 62, 0.1% (no support) 9874-24 [\$\tilde{5}\$-\tilde{1}\tilde{2}\tilde{2}\tilde{1}\t	363	\$11-9539	Simetallic, 52.4, 47.67 (no support)	0.27	0	0		0	
9874-24 [\$\tilde{5}\tilde{7}\tilde{4}\tilde{8}\tilde{8}\tilde{1}\tilde{9}\tilde{9}\tilde{1}\tilde{9}\tilde{1}\t	354	6187-17Œ	Irimetallic, 32.9, 62, 0.1% (no support)	0.92	→ 'S	60	•	16, 15, 16	
6616-104 Bimetailie, 26.2, 73.8% (no support) 6749-161 Bimetailie, 55.1, 34.9% (no support) 6649-161 Bimetailie, 53.7, 46.3% (no support) 10.69 10.260-758 If Pt/R-8 Alg0, support (control) 10.280-790 Bimetailie, I, If/R-8 Alg0, C.42 10.280-790 Bimetailie, I, If/R-8 Alg0, C.42 10.280-790 Bimetailie, I, If/R-8 Alg0, C.43 10.280-804 Irimetailie, I, If/R-8 Alg0, C.43 10.290-804 Irimetailie, IIII IIII IIII IIII IIII IIII IIII I	365	72-1285	IS 24/R-8 Al_03 support (ref.)	0.42	27,		式	15, 73, 72	
6749-161 Simetailic, 65.4, 34.9% (no cupport) 56.6-101 Simetailic, 53.7, 46.3% (no support) 10260-758 If Pt/R-8 Alg0, support (control) 10280-792 Simetailic, I, If/R-8 Alg0, 10280-792 Simetailic, I, If/R-8 Alg0, 10280-792 Simetailic, I, If/R-8 Alg0, 10280-795 Simetailic, I, If/R-8 Alg0, 10280-795 Simetailic, I, If/R-8 Alg0, 10280-795 Simetailic, I, If/R-8 Alg0, 10280-804 Irisetailic, II If/R-8 Alg0, 1028	366	9646-:04	Bimetailie, 26.2, 73.8% (no support)	66.3		0			
56-6-101 Simetaille, 53-7, 46.35 (no support) 0.48 27, 27, 21 56, 52, 51 74, 10.260-752 15 ft/k-8 Alg0, support (control) 0.41 14, 12, 12 31, 23, 22 17, 10.280-792 Simetaille, 1, 15/k-8 Alg0, 0.41 14, 12, 12 31, 23, 32 17, 10.280-792 Simetaille, 1, 15/k-8 Alg0, 0.42 21, 21, 22, 22 54, 13, 13, 14, 15, 13, 14, 15, 15, 13 15, 10.280-795 Simetaille, 1, 15/k-8 Alg0, 0.42 25, 15, 13 45, 25, 35 68, 10.280-804 Frisetaille, 1, 15/k-8 Alg0, 0.43 18, 18, 18, 18, 18, 18, 18, 18, 18, 18,	367	191-6129	Simetailie, 65.4, 34.97 (no aupport)	==	0	-		0	
10280-758 15 ft/R-8 Alg0, support (control) 0.48 27, 27, 21 56, 52, 51 74, 10280-792 816staille, 1, 15/R-8 Alg0, 0.41 14, 12, 12 31, 23, 22 47, 10280-792 816staille, 1, 15/R-8 Alg0, 0.42 12, 13, 11, 13, 11, 29 54, 10280-755 816staille, 1, 15/R-8 Alg0, 0.42 25, 15, 13 45, 25, 35 68, 10280-804 Fisetaille, 1, 15/R-8 Alg0, 0.43 18, 18, 18, 18, 18, 18, 18, 18, 29 54, 39, 29 64,	38	101-9-99	Simetailie, 53.7, 46.3% (no support)	0.49	0	_		0	
10280-79C Bicerailic, ,	363	10260-758	15 Pt/R-8 AlgOs support (control)	0.48	27.	56.		بخ	
10280-790 Bicerailie, i, if/R-8 Al ₂ 03 C ₂₋₄ 2 18, 13, 11 33, 31, 29 54, 10280-795 Bicerailie, i, if/R-8 Al ₂ 03 C ₂₋₄ 3 21, 23, 22 51, 50, 48 76, 10280-795 Bicetailie, i, if/R-8 Al ₂ 03 C ₂₋₄ 3 C ₂₋₄ 3 (C ₂₋₄ 3 18, 18, 18, 18, 18, 18, 18, 18, 18, 18,	8	10280-790	Biostailic, 1, 15/8-8 Al 202	→	2	=		=	
10280-735 Bimetaille, 1, 15/R-8 Al ₂ 03 C.43 21, 23, 22 51, 50, 48 76, 10280-735 Bimetaille, 1, 15/R-8 Al ₂ 03 G.42 25, 15, 13 45, 25, 35 G.8, 10280-804 Friestaille, 1, 1, 15/R-8 Al ₂ 03 G.43 18, 18, 18, 18, 16, 44, 33, 39 G.4,	371		Bicetailic, 1, 15/8-8 Al 203	C-+5	Ξ.	ដ		~	
10280-804 Trimetallic, 1, 15/R-8 Algos 0.42 25, 15, 13 45, 35, 35 68, 10280-804 Trimetallic, 1, 1, 15/R-8 Algos 0.43 18, 18, 18, 18, 18, 18, 18, 18, 18, 18,	32		Bietaille, 1,	C.43	73			, X;	
10280-804 Trimetailic, 1, 1, 1\$/R-8 Al20s C.43 18, 18, 16 44, 39, 39 64,	373	10250-795	Bisetalite, 1, 14/R-8 41203	27.0	5.	÷5.		23	
	35	10290-804	Trimetailic, I, I, IF/R-8 Algos	0.43	80	<u> </u>			

a) LHSY = 200. b) Same charging unight as for catalyst 9874-24 (ref.)

Table 66 (Contd-5). MCH DENYLAUCENATION WITH VARIOUS CATALISTS IN MICHR: RUNS 263-450

٩	621.5	Catalyst		S ECH C	f UCH Convarsion, "F	
9	Ko.	Description	#t, 9	299	252	2+8
ا ا	ישטע שער	Bi - in [5/2.9 1] . 0. encoot	0,42	21. 21. 73	40, 37, 39	S 88 8
3 5	200 0000	Simple life 1 1/70-8 Al. (), stranger	27.0	<u>.</u> ∞		65, 65, 59
3 5	20-70701	of persons and annount free.	0.43	16, 24, 24	58, 50, 49	ಚ, ಚ, ಬ
375	10230-814	A meta(b)/type autoport (confre)	8.0	34, 28	51, 53, 54	181, 77, 77
37.	813-08201	12 metaic)/type (support (centrol)	0.7	8 8	60, 58, 53	85, 85, 80
5 5	10200	_	0.74	ਲ ਲ	ģ	(92), 87, 84
3 5	10280-810	A metaie / type support (confre)	2.0	29, 27	55, 56, 56	
£ €	10280-815	_	0.73	33, 32	5	
1383	10280-815	to metal9)/type i support (contrai)	37.0	સ્		=
384	10280-834	15 meta! /30, type support - 20, type & binder	6.73	23, 24, 23	53, 47, 47	66, 71, 66
8	10780-815	(setal/type support (cuntrol)	0.74	31, 31, 33		æ,
386	10786-838	25 metal /80% type i support - 20\$ type 6 binder	5.0	<u></u>	55, 55, 51	13, 73, 62
38	10280-830	17 retal/805 type I support - 205 type 6 bloder	0.67	23	52, 52,	Ę
388	00260-830	Ze metal/typo surport	2.0	%	2,	≎
33	10780-815	metel/type	0.69	25, 36, 33		85,
8	10780-813	_	0.59	ន	56, 61, 61	85, 89, 85
33	10280-810	-	2.0	22	8, 15,	8
392	10280-310	4. metal/type i supports)	0.7	73, 35, 34	76, 62,	£
393	5874-24	1, Pt/R-8 Alaba support (ref.)	CC	7,	2	3
Ř	10280-816	4; metal/type i supporta)	8.0	28, 33, 32	63, 89,	Š,
38	10280-816	(/ metal/type supports)	0.7	ੜੱ	8	30, 68, 34
336	6546-1436		18.0	0	<u> </u>	-
387	10230-897	_~	0.56	75, 23, 27	80, S6, S1	82, 89, 73
		40£ type I support (2).				
		2. type 6 binder				
		7000				

Unneutralized ispregnate (hydroxids). Calcined in sir I hr at 965 F.

Acid i neutralized icpregnate. Acid 2 neutralized inprognate. Acid 3 neutralized icpregnate. Acid 4 neutralized icpregnate. ಕರ್ಶಾರ್ಥ ಚಿ

Acid S markedized tepregate.

Table 66 (contd-6). MCH DEHTIROGRATION VITH VARIOUS CATALIYSIS IN MICHR: NUMS 263-450 75, 71 87, 76 7, 75 81, 75 85, 88 8 23 83 2 **28** Z 219 ä 25, . . 8 6 ឌ ಡೆ ಹ <u>د</u> . 8, 2 a, \$2° 73 rir: ឌ 8, ≂ ಚ S MCH Camerefen, X 28 28 888 53 **\$** Ħ ₩ 25 8 52 757 2,8,0 62, 51, នេះនេះនេះ 8, 53 × **휴** 뗞 × 52,53 χ, £8, ίζ, 3, 2, 3, 62, ᅉᇔ S, 8 23 22 23 22 22 22 22 27 22 ក្នុងក្នុងក្នុ 2, 瓦 22,22 :3 జ్ బ్ 32, 652 **≈**′≈ ž # X # ť ≘ೆ ನ 27, 33 #3 ť 0.54 0,57 3,71 \$ 12.00 \$ 12.00 D. 22 다. 다. 다. 13.0 25 metal/type i support (spheres)
14 25 metal/type i support (spheres)
15 lessail/type i support (spheres)
16 liseatal/type i support, 155 type 6 binder
17 metal/275 type i support, 155 type 6 binder
18 metal/275 type i support (1),
17 metal/275 type i support (2),
27 metal/275 type i support (2),
27 metal/275 type i support (1),
27 metal/275 type i support (1),
27 metal/275 type i support (2),
Trinatallic, 53.5, 2, 44.5% (no support) metaible]/39/ support type i (1), 39% support type ! (2), 39's support type 1 (2), 20, type 6 binder 20, type i support (1), 67, type i support (2), 135 type 6 binder 15 PUR-8 41203 support (ref.) 25 setal:}/396 support type : (1), Catalyst type I support (!), type I support (2), If Pt/R & At., G. support (ref.) If setal/13r type I support (!), Description 67. type I support (2), 22% binder type 6 20, type & binder 2 mtel/40, εí 12 355-09201 \$874-24 102-00-508 10280-350 10280-324 9274-24 10280-92C 965-08Z01 1815-0820 10280-914 10280-928 1026-1320 6646-88 1624 - 48C Catalyst Š S Ę. 398 청중호유효혹중 9 \$ \$ 8 ₽ = **21**

s) Prepares for beach scale evolution. b) Carbonated estal laprognate.

Calcined in air at 1112%.

Blackellic, 10, 42/type i support

10290-102C

Property for beach state evaluation,

Carbonated metal impregnate, Calcined in air at 1112 F.

Table 66 (Comtd-7). MCH DEHTTROGENATION WITH VARIOUS CATALYSTS IN MICTR: RUNS 263-450 312 K 88 ಜ **∓** ≅ 2 8228 **₹** % # 47, 47, 91, 80, 1 25.55 ŧ 8 జ ï **ಬ್ರೆಜ್ಜೆಜ್ಜೆ** 8, ₹, 8 4 ង្គ្រំង 83 ឌ **ಟ್ರಪ್ಪಜ್ಜಿ** ಜೈ œ. 8 1 MC Corrorator. **8** 2 ij S 2 \$ 42 + 22 **33** 83 22 22 23 35. 2,2,2,8, 2,2,2,8, SS SS 37, 45, 13, 45, ង់ខ្ល 52, ಜ್ಞ ٣, ٩, ٩, ١, 53 ij ŝ Ę 67 33, ¥.4.4. 53 ន្ត នេះ នេះ នេះ 85, 28 19, 19 18, 28, 28 3 5 22 35 8 7 27, 293 27, 30, ខ្លួន ଞ୍ଚ 86 ₹, 27, 3,53 ជំង្គីង 뜩 줘 ε<u>.</u> Κ΄ ε. Ŕ + ක<u>ී</u> 9.5 3.51 0.5 0.5 0.57 0.0 0.0 0.0 0.0 50000 50000 50000 50000 2f matel/805 type I support5//205 type 6 binder 6f matel/type 6 support5 57 matel/type 6 support 57 matel/type I support 8 matellie, 2, 6//type I support 8 matellie, 5, 6//type I support 2f matelc)b)/3sf support type [[], 3sf, support type [(]), 37 mer., (0, type i mur., 20, type i mur., 20, type i support (1), (0), type i support (2), (0), type i support (2), (0), type i support (2), Catalyst 39, support type I (2), 27, blader type 6 metal/type 6 supportu)
metal/40/ type 1 support (1), is estal/39, support tym ! (1), 39% support type 1 (2), 22, binder type 6 metal/39, support type [[]], Description 22, binder type 5 26, type 6 binder सं र्य 7 10280-33v 10280-1003 10280-980 10230-98€ 886-08201 9874-24 1,780-1006 10,230-1048 :0280-106C 10280-1558 10280-989 0230-1C3A 10780-1078 10280-102F Catalyst Ş **£**13 \$ **415** 415 5 Š **2358** 222222

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CANADA MANAGEMENT CONTROL

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Table 66 (Contd-8). WER DESTUROCEMENTION WITH VARIOUS CATALISTS IN MICHR: RUNS 265-550

. 3-

5	Cain lyst	Catalyst				34	4 ICH Comerator,	# SR, F		
<u>\$</u>	Ko.	Description	4,0		299		722		8	
55	3201-08201	Simitalife, 5, 26/kyps ! support	0.46	z	24, 21		88.	æ.	51, 52	
3 5	בהיסת-ותש	Simetalite, 3, 6, ftype	3		22, 23	₹.	35, 25		8, 8	-
<u> </u>	מנטן המאס	Binetallic, 6, 6,/type	8		; 0 ;				9	
? :	OC01-2070	presenting by type I support	_			_	28, 23		ص م	
3 6	201-130	It It it is a support (nee ref. prepa.)					÷, t		۲. ت	
454	3874-1378	S. metal, S. metal/type support		a a	39, 37	<u>ಫ</u>	72, 62	8.	5: 5:	
÷	3074-176	-				_	€, 43, 49		8	
25	05-63-20		200		0		0		9	
437	6749-48		9.0	_	0		0		0	
<u> </u>	6749-338	Binetallic, 38, 52) (no support)			*	9	٠, س	21. 2	0, 19	
1396	0301-08201	S. metal/802 type i support, 202 type 6 blinder		5,	45, 43		3 6		8.	(q. 8
100	JH 0280-104C	5, metal/40, type (suggert (1),		46, 4		83,b)	67,	66 89,b)	89, b) 88, b)	(488 a
		40, type I sepicat (2),			•		•	•	•	
		20/ type 6 binder								
25	3001-00ZUN(5)111	55 metai/80; type I support, 20/ type & binuin			8 8	×,	(3), 43	_	75, 6) 30, 6)	=
1 (42	JE03-18201	Binetailic, 2, 5,/type I support d)					55, 54		e, E	
£ 3	2874-90		10.0			-		. 85	200	
111	3874-193A				25, 28	2	52, 48	72. 1	2	
£ 5	10280-1093	3, metal/40, type i support (1),		31, 2	25, 24		61, 58	8,	7, E3	
		46, type I support (2),				-				
116	out uscus					_				
	1000					_				
-	SC1-00701			Ŕ	ដ	_	22 25		ල. ස	
?	BC01-026	4: metai/support type 6				_				
**	10280-1040	Se metal/40; support type I (I),	59.01		22, 25	8	51, 46	9, 6	æ 8	
		40' support type i (2),								
3	9501-0920	55 mtal/	r.	.; &	28, 23, 26		45, 42, 43		69, b) 50, 7	
]-					-	_		7		

Carbonated satal impregnate.
Includes substantial amounts of benzone.
[ENY = 50.
Calcined in air at [112]. ಎಪ್ಪ ಕ

on a the Madding

Table 67. MCH DEHYDROGENATION WITH VARIOUS CATALYSTS IN MICTR: RUNS 484-572

Period: December, 1967 - Fetruary, 1998.
Conditions: 10 atm pressure; catalysts reduced in hydrogen at 196°F,
-AL samples formally taken at 3-, 8-, and 13- minute
(cormitin at each temperature.
0-9 microtactive district with 1-1 mi quarte chipe| THEY = 100 (catalysts 4-4 quarts 10-20 mesh).

		THOM . Its Containate an	of diest	10.50 met).	
20	Cotalyet	Catalyet		4	MCH Conversion	, 7
40.	No.	iw teriftica	VI, CE	64.2	152	342
5	10454-118	Programmy	0.275	10, 4, <	7, 7, 7	8, 5, 5
lid)	10/A0-111A	20% metal/type 1 Juggeret	0.8.9	10, 9, 9	9, 2, 9	12, 11, 11
486	103-0-1116	in wtal/type 1 mills rt	0.1 m	18, 14, 14	25, 21, 47 25	33, 31, 29 ⁶³
48 7	10,40-117A	rs notal/406 type 1 august (1) /406 type 2 august (2) /406 type 6 million	ა. ჯა	.6, 24, 25	55, 56, 52	61, 85, 76
rw.	10280-1178	b# metal/50% type 1 support (1) /50% type 2 support (2) /30% type 6 support	0.334	25, 26, 4	%, % ()	60, 19, 19
Lity	10260-1170	fristailis, 2,2, 25/type 1 empera	C.821	16, 12, 15	28, 27, 27	27, ⁶⁾ 18, 11
49 0	9874-139	16 Pt/R-8 type AiaCs	0.626	22, 21, 25	47, 49, 47	76, 75, 67
491	10290-118	Pinetallie, 2, 69/type 1 support	0.465	0, 0, 0	0, 0, 0	0, 7, 0
133	10280-77A	25 matel/405 type 1 support (1) /405 type 1 support (2) /405 type 6 support	0.501	22, 51, 21	57. 58, 55	82, 1654, 17
493	6646-18	trimitallio, 55.4, 2, 64.65	0.465	0, 0	0, 0	0, 0
494	6749-26	Trimetallic, 5:8, 1:4, 94.8\$	0.526	0, 0	0, 0	9, 0
195	6749-55	Trimetallic, 2.7, 4.2, 95.1\$	1.43	0, 0	0, 0	0, 0
696	6646-39	Bimetallio, 48.2, 51.85	0.215	9, 10, 0	5, 6, 5	6.3. 3
697	67 by - 162	Bluetallic, 68.4, 51.6\$	0.824	0, 0	0, 0	0, 0
198	6749-108	Trimtallic, 35.8, 6.2, 60%	0.611	3. u	۵	a, A, 11
443	6646-1990	Binetallic, 3, y5\$	0.955	0, 0	0,0	c, o
500	3603-165	Binetailin, 30.9, 61.5%	0.576	0, 0	0,0	9, 7
901	9874-159	18 Pt/R-8 type AlgOs	0.626	b)	6)	•)
205	10760-119A	14 metal/type 1 (control) support	0.612	25, 25, 22	46, 52, 52	75, 79, 73
503	10260-1190	Nimetellie, i, 2%/type 1 s.pport	0.721	54, 23, 26	56, 52, 55	13, 18, 15
90% 905	9874-139 102 9 0-121	is Pt/R-8 type aimes (Prf)	0.426	29, 24, 24	50, 52, 19	16, 76, 72
906	10260-121	A% metal/type 1 support Bimstallic, 1, 1%/type 1 support	0.721	80, 27, 26	55, 55, 54 48, 48, 51	19. 62, 19
907	10200-1190	blastellia, i, M/type 1 support	0.142	20, 22, 22	51, 49, 14	11, 15, 11 65, 65, 64
508	10280-1198	1% metal/type i support	0.694	24, 18, 20	44, 45, 40	61, 60, 56
309	10289-121	45 metal/type 1 support	0.724	24, 26, 29	30, 55, 55	82, 85, 82
310	10280-124A	25 Mts1/type 1 support	0.573	22, 22, 25	33, 46, 31	18, 19, 16
311	10200-1268	48 mate:/type 1 support	0.632	21, 26, 22	51, 51, 51	76, 75, 7h
312	10:50-1190	Bimtellic, 2, 18/type 1 support	C. 724	23, 24, 22	10, 51, 18	73. 75, 76
51.5	10280-1199	Bimetallio, 5, 15/type 1 support	0.746	23, 24, 22	57. 36, 51	95, 79, 73
326	9674-199	15 metal/8-8 type AlpCy (ref)	0.434	27, 24, 25	20, 53, 51	71. 75. 70
519	10200-1153	Piertellie, 2, 25/type 1 support	0.882	35, 23, 24	58, 53, 55	80, 84, 86
316	10280-1191	bimetallic, 5, 35/type 1 support	0.159	25, 26, 22	56, 57, 55	70, 02, 01
517	10080-130A	Nimitallie, 1, 'Waype 1 support	0.682	19, 19, 17	14, 10, 10	56, 17, 56
51 d	10280-1208	Nimitablie, 1, 24/type 1 support	0.642	16, 20, 20	39, 38, 36	46, LT, 48
119	10560-1190	Biumtallio, 1, 35/type 1 muppart	0.751	17, 24, 19	38, 39, 36	93, 58, 676)
520	10380-13-6	is metal/type 1 support	0.770	0, 4, 2	:3, 10, 9	19, 21, 21
521	705Q0+7%0#	Simmtellia, 2, ly/type support	C-72)	25, 20, 20	42, 47, 41	61, 61, 99
529	10590-7504	bimetallia, 3, 15/type 1 support	0.720	20, 22, 20	-6, b6, b5	12, 17, 15
323	10240-1300	Nimetallie, 3, 5%/type 1 support	0.800	16, ly, 71	45, 39, 36	55, 50, 50
231	10260+120H	Firmtallio, 3, 25/type 1 support	0.761	23, 21, 10	45, 46, 47	69, 61, 60
525	9474-159	1 Pt/R-8 type Alpha (ref)	0.453	23, 21, 20	U, 17, V	72, 69, 70
526	10260-129	b\$ metal/type 1 supportald1	0.691	37, 32, 29	36, 36, 11	19, 11, 16
521		ph weres/sale 1 enticares	0.673	25, 26, 26	57, 57. 9	72, 73, 77
528	10590-1557	Trimetellia, 1,1, 19/type 1 support	0.748	27, 26, 26	56, 54. (48)	66, 73, ft
						(Cust (nu n))

Table 67 (Contd). MCH DEHYDROGENATION WITH VARIOUS CATALYSTS IN MICTR

7,0		Cotalyst	 	•	It'il Conversi	on, "F
	No.	Dracelption	VI, E	6ir	752	Alig
349	1	Trimetallie, 5,5, 5%/type 1 support	0.645	32, 24, 25	16, 32, 19	6), 67, ()
330		fri-mtallio, 3,1, 3%/type 1 support	0.748	23, 22, 23	51, 40, 48	34, 37, 37
23/	1	18 Ps/8-8 type LigCa (ref)	0.630	15, 26, 26	48, 47, 49	12, 14, 12
332		Trimetallie, 1,1, 15/1990 i support	0.7%	22, 20, 22	19, 16, 16	62, Cb, 62
333	1	the manney with the thing of any last	0.790	27, 29, 26	j 46, 45, 47	65, 52,-) 44
334	10760-1728	Trimetallie, 3,3, 15/type 1 support	0.770	79, 28, 27	52, 52, 51	61, 70, 69
333	10560-1558 10560-1558	Trimetallis, 1,1, 35/type 1 support	0.155	12, 2), 23	45, 64, 64	39, 0) 35, 54
3 % 337	10500-1554	Trimetallie, 5,1, lf/type 1 support	0.771	26, 24, 23	32, 32, 36	n, n, 12
330		(frientallie, 5,5, 25/type 1 support	0.772	22, 21, 25	10, 14, 15	66, 67, 67
150	10140-13UM	10201-1191*)	0.723	26, 26, 22	96, 55, 57	80, 76, 76
منرا	10260-150	1026)-119[*]	0.742	24, 26, 23	33, 33, 34	77, 63, 77
341	10-6 - 1300	16/00-1900	0.750	23, 21, 20 22, 22, 21	55, 49, 49	76, T2, T2
34	10,90-127	18 metal/type 1 support	0.793	10, 22, 25	49, 40, 50 47, 47, 46	78, 75, 74 70, 70, 69
٧,	10240-1501	b\$ metal/type 1 support*)	0.612	30, 24, 22	47, 45, 45	70, 10, 10
344	10380-1364	26 metal/act type 1 restore (1)	0.392	13, 11, 10	10, 21, 16	10, 10, 10 10, 39, 39
1		26 metal/aC4 type 1 memors (1) /kO4 type 1 support (2) /aU4 type 17 binder		.,, .,, .,	,~, -,, ~ 3	~, ,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,
545	9874-139	IN PI/N-B type AleCo (per)	0.695	30, 24, 36	50, 44, 48	67, 71, 68
346	10280-1268	Similar to 10280-1264 1	0.539	15, 18, 11	31, 113, 24	60, 34, 33
347	10560-1750	Atmetalite, 6, 6%/type 1 support	0.660	0, 0	0, 0	0, 0
310	7€1%-80	≥\$ wtal/type i support	0.419	20, 11, 23	51, 48, 48	78, 77, 76
749	10000-1944	Mumicallio, 3, 3%/type 1 support	0.744	20, 24, 26	10, 19, 50	39, 56, 36
330	100A0-13eB	Bimetall's, 5, 15/type 1 support	0.716	20, 24, 26	5h, kg, kg	71, 69, 75
351	10200-1524	Bimminilin, 2, 65/type 1 support	0.729	20, 19, 26	43, 48, 45	60, 70, 64
310	10500-1259	a.aetailis, 9, 65/type 1 support	0.717	0, 0	0, 0	0, 0
255	10200-1314	3% matel (3% type 1 supropt (1) 3% type 1 support (2) /10% type 6 binder (12% type 15 binder#13) 937%-139*/	0. 498	19, 21, 19	54, 51, 50	76. 17. 19
354	10060-1604	9379-1991 (129-1979-15 Bloder-177)	0.404	23, 19, 26	47, 44, 45	69, 64, 66
555	10260-1428	98° -90°)	0.406	85, 80, 19	45. 61. 42	64, 67, 67
774	10860-1909	36 mets]/hOS support type /hOS support type 1 /205 support type 6	6. <u>19</u> 6	87' Le' 8T	47, 40, 40	41, 75, 71
357	10060-19-0		0.719	31, 21, 52	35, 34, 31	78, 74, 10
558	987h-7	9874-7	0.758	23, 26, 22	32, 49, 51	16, 18, 75
339	9076-139	15 Pt/R-8 type AlaGa (19f)	0.44	19, 24, 25	46, 46, 40	70, 12, 12
940	102 0 0-1518	95 tetal/946 type 1 ampoint (1) /995 type 1 emport (2) /256 type 6 binder#]]]	0.562	34, at, at	55. 51, 90	60, 19, 17
961	10590-1330	10260-1530 ^f)	0.571	24, 22, 21	54, 50, 49	19, 14, 13
762	10260-1554	76 math/606 type 1 out mark (1) /606 type 1 r () try (2) /606 type 6 (n.dr)	0.481	31, 85, 85	51, 50, 50	16, 73, 16
363	10080-1539	ys metal ³ /AOS type 1 support (1) /AOS type 1 support (2) /2OS type 5 "inder!)	0.675	19, 19, 24	45, 41, 4n	15, 60, 57
34 4	10260-1330	36 detai/606 typ. 1 support (1) /606 type 1 support (2) /206 type 6 binier)	0.493	30, 24, 92	50, 47, 46	76, 70, 74
345	9614-1994	48 mais /type 1 support	Ú.835	20, 30, 21	50, 51, 51	78, 8c, 76
365	9874-199C	his metald)/type 1 supporth(c)	0.752	22, 18, 25	W, W, W	n, n, 69
767	9876-1990	## mrel/type 1 nappore #1 a)	0.741	21, 21, 22	56, 54, 51	15, 17, 16
760	9876-2008	iff meat? /supe t support	0.560	36. 26, 21	51, 50, 50	80, 83, 19
369	9U71-830C	45 metal ^{d)} /type 1 suppose ^{d)}	0.948	24, 21, 25	54, 55, 59	Se, So, Re
370	9674-199C	Repost of Run 366	0.7.8	43, 80, 25	37, ×, ×	80, 19, 19
571	70360-191B	56 metal ^{h)} /type 1 support 56 metal ^{h)} /type 1 support	0.655	24, 21, 23	45, 44, 44	36, 50, 99
374	10260-1414	39 betai //spe 1 support	0.193	20, 21, 21	50, 47, 45	73, 67, 69

^{72 10200-1818 | 96} metal "/sppc 1 support 0.595 | 20,
Includes substantial manufact beneme.
GC bised line overheades, sampling evacting of the products.
MarTied 109879 |
Ourbonated impregnate.
Albalime type 6 binder.
Support marfiel at 111279.
Inclusion .f 128 established metal.binder.
Impregnate as.a neutralised.
Support marfied at 111279 before impregnation.
Support marfied at 3217 before impregnation.
Support marfied at 3217 before impregnation.
Support marfied at 3217 before impregnation.

(Continued)

Table 68. MCH DEPATROGRAFION WITH VARIOUS CATALYSTS IN MICTR: RUNS 573-676

Period - March to May, 1968	as: 10 stm pressure. Catalysts reduced in No for 30 min st 796's.	CLC samples normally taken at 3-, 8- and 13- min operation of	each block temperature. Granular catalyst volume: 0.9 ml	catelyst diluted with 1,1 ml querts chips. Lined estalyst tubes	either empty or packed with quarts chips, as indicated.	ZHSV = 100.
	Conditions:					

g	Catalyst	Catalyst		38 5	\$ MCH Conversion, T	£ 4
Š	.te.	lescription	Se ight	295	lsi.	842
225	821-1/85 5E1-1/85	is filling R-8 type kiggs (ren)	62.53	19, 18, 23	0, 0, 12	56, 69, 67
55		of Pt/type (0.616	į Kį	51, 56, 49	76, 72, 71
576	10280-1430	45 Pt/type i support, 11327-1-1/2 hr air 45 Pt/type i support, 12377-2 hr air	0.687	2, 23, 22	51, 49, 47	13, 72, 72 15, 72, 72
578	10280-143E	£.	229.0	É	41, 36, 34	51, 53, 51
£ 5	10280-144	35 metal/type I support, stabilized	œ 6	Z Z	43° 46° 48	2, 2, 2, 2, 2, 2, 2, 2, 2, 2, 2, 2, 2, 2
3	9874-139	IS Pt/809 R-8 type Alga (ref)	0.435	25.5	\$ \$ \$ \$ \$ \$ \$	2, 2, 2, 2, 2, 2, 2, 3, 4, 4, 4, 4, 4, 4, 4, 4, 4, 4, 4, 4, 4,
*	19780-151	Tube No. 1, "25 metal/305 type I support (1) ^{b)} 405 type I support (2)	0.739	25, 25, 26	71, 71, 71	87, 8S, 87
8	151-08201	207 type 6 binder Same as above with quartz filler	622.0	42, 37, 36	76, 75, 77	96, 97, 97
3	251-08201	Tube No. 2, -25 metal/405 type I support (1) ⁶⁾ 405 type I support (2)	0,406	32, 28, 23	83, 81, 81	78, 77, 75
뚌	10280-152	Same as above with quartz filler	907*0	41, 41, 40	71, 71, 75	97, 96, 95

some benzene formed with teluene.
 Empty tube, catelyst on well; 0,002 in, thick well, 1/4 in, 00,
 Empty tube, catelyst on well; 0,028 in, thick well, 1/4 in, 00.

Table 68 (conte-1). How destrocaraction with various catalysis in Michel Ruis 573-676

22 23 ۴ 5 to Si 325228 95 た 礼 和 乾 82 Continued) 80, 77, 95, 95, 95 79, 77, 94, 93, 5. 13. 0 83, స్ట్రే **జ్** 8 4 2 3 8 5 82 o. 55 8,2, ۴. ឌ **%** ≠ 8 6 \$ MCH Conversion, R sı = 57 傷 守 弘 亀 4: 5: 特的作品 67, 63, 63 65 7 . . . တ တ် " સું સું સું સુ 22 ೫೭ Į, Ę 752 8 2 2 2 E, డ్డి జ్ **នឹក់សំសឹត** ಚಿ.ಕ್ತ 5 7 13, 12, 23 23, 23, 23 23, 23, 23 24, 23, 23 27, 23, 23 27, 23, 23 8 2 3 8 88 2222 27 8 o 0 ក់ពីជំនំ សំភំសំសំ 8 8 7 7 × ន ឌ័ឌ Rì జీ **662** ċ × ន្លង 23. ₹, Teight 0,683 0,583 0.435 0.778 0.729 0.711 0.128 0.25 0.25 0.25 0.707 9.48 9.48 9.48 9.48 9.48 0.717 0.128 ន្ទ 8 Empty tube, catalyst on wall; 0,028 in. thick wall, 1/4 in. 00. Tube No. 6, coated with No. 18 binder, pistinized Tube No. 3, ~26 Metal/406 type i exprort (1)2) 4:36 type I aupport (2) [ube No. 7, -26 Hetel/405 type i support^{8]} 45 Pt/type 1 support (chasically reduced) 40% type 2 support 20% type 6 binder Tube No. 5, Same conting as tubo No. 3 Tube No. 4, State conting as tube No. 3 20% type 6 binder Same as above with quartz filler State as above with quartz filler Same as above with quartz filler Same as above with quartz filler 15 Pt/UGP R-8 type AlgGs (ref) 15 Pt/type I support, 248°F dried 15 Pt/USP diluted to 4 in. volume Cttin **Greatistis** if Pt/UOP R-8 type Algos (nef) Report of run 575 Report of run 576 Repeat of run 578 Report of run 577 Report of run 561 10280-14Œ OC+1-08ZC1 10280-1430 Catalyst 10780-158 13780-158 10280-143A 10280-1435 10280-1330 10280-158 10280-165 10280-158 10280-158 521-1286 E+1-0620 10280-165 9874-139 10280-165 9874-139 ė 88 88 28 88 88 592 S **5888** 9

5	3	Catalyst				E ST	KKH Conversion,	چ بر	
<u>.</u>	₩c.	Becciption	10 ight	962			752	278	
888	10280-167 10280-167 10280-168A	Irisetaliic, 4, i, 65/typs i support 45 Meizi ⁵)/typs i support Bisetaliic, 4, 85/typs i support	0.780 0.761 0.754	14, 12, 10 15, 14, 13 14, 12, 12	2000	8 8 8	27, 21, ⁴) 18 31, 24, 21 73, 26, 25	30, 21, 18°) 44, 40, 3) 35 46, 38, 36	(8 3 g)
88	10280-170	Tube No. 10, 35 Pt/405 type i support (1) ^{b)} 405 type i support (2) 205 type i support (2)	85.0	22, 22, 22	8		47, 50, 48	65, 67, 66	9
803	10280-170	Same as above with quartz filler	0.083	25, 23, 24	5 24		59, 54, 52	8° 80° 80	8
5	10280-170	Tube No. II, 35 Pt/405 type I support (I) ^{b)} 405 type 2 support (2) 205 type 6 binder eith quartz filler	0.20	8 ° ° ° ° ° ° ° ° ° ° ° ° ° ° ° ° ° ° °	ਲ .*		85, 87, 80	97, 99, 36	<u> </u>
619	10280-1338			25, 21, 22	2		3), 4C, 46	15, 73, 12	~
5		Of type I support	0.514	24, 24, 23	ş 23	6 3	50, 45	13, 14, 73	
613	10280-1470	Same as above with quartz filier	925*0	22, 21	21, 22	8	34 °6+	14, 71, TE	۔۔۔۔
A 22 25 25 25 25 25 25 25 25 25 25 25 25	9674 -138 9874 -170 9874 -153A	<pre>18 Pt/UCP R-8 type Ai_20_g (ref) Tube No. 11,b) ref to Run 610 Bimstalile, 9, 95/type I support</pre>	0.422	888	ខ្លុំ ដូ ខ្លួំ ដូ	8,8,3	50, 47, 46 69, 67, 67 40, 34, 34 ⁸ .	69, 69, 70 84, 85, 85 (17),(13),(13) 35, 34, 35°	35.03 35.03 35.03
ಕಿತ್ತುವಿ	Some Somers Eapty tube, Bracketed my Ispregnated	Some benneme formed with tolumne, Empty tube, catalyst on well; 0,028 in, thick well, 1/4 in, 00. Bracketed numbers benzans, unbracketed numbers total conversion of NCH to benzans and tolumne, imprepasted in chaiste form.	of KCH &	o benze	3	k te	ğ	(Cantigued)	3

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Table 68 (Contd-3). MCH DEHTTROCEMATION WITH WARIOUS CATALISTS IN MICTR: RUNS 573-676

S.	చ	Catalyst			~	≸ MCH Conversion,	Ę		4	
કુ	Ko.	Becription	Te ight	293	_		752	}	873	
219	9874- 173	<pre>iube No. 9, 35 Metai/405 type I support (!) 405 type I support (2) 205 type 6 binder Filled with quartz</pre>	0,335	23, 25, 78	5, 38	S '63 '65	, et	1	70, 65, 62	29
	£21- 1286	Tube No. 8, 3% Meta!/40% type I support (i) 40% type I support (2) 20% type 6 binder Filled with quartz	0.1451 40, 37, 36 83, 78, 78 (28,(12),(10). ⁸⁾ 95, 97, 65	5	36 %	ដ	ه. د	<u> </u>	(2), (12), (16 95, 97, 65	((Ø) 2) 65
619	9874 -174A	9874 -1744 Tube No. 12, 35 Metal/40% type 1 support 40% type 19 support 20% type 6 binder Filled with quartz	0.125	42, 32, 30 m, m, m	8	,	é é		16 18 58	~
620	10280-173	620 10280-173 Tube No. 8, of Run 618, filled with quartz	0,145	% %	ž 5	8	52,	= = =),(66 7, S7	28, 25, 24 80, 57, 55 (14),(66),(69)*)
22	10280-1740	10280-1746 25 Pt/405 type I support 405 type IS support 205 type 6 binder	0.525	2, 24, 22	22 '1		53, 50, 48		78, 76, 77	t :
223	I0280-1740	3% Pt/40% type i support 40% type i3 support 20% type 6 binder	0,5185	2, 2, 2	2	55, 52, 52	25 25		82, M,	Ħ
625 625 625	3521-08201 10280-1744 10280-1755	Tube No. (2 ^{b)} , of Run 619 45 Pt/type i support (isprag. 18 heurs) 45 Pt/type i support suffled at 11(2°F, in air	0.126 0.705 0.672	2, 23, 23 25, 25, 25 22, 22, 22	5 B B	25, 25 25, 25, 25, 25, 25, 25, 25, 25, 25, 25, 25, 25, 25,	ស្នួនូ <u>ខ</u> * ខ.ខ.	्रक का स्टब्स	63, 62, 62 81, 78, 78 79, 77, 76	55 78 75
2	Bracketed mu	B) Bracksted numbers benzens, unbracksted numbers total conversion of MCH to benzens and tolusns.	of ECH to	penze	15	15	8		(Continued)	(Da)

8) Bracketed numbers benzene, unbracketed numbers total conversion of MCH to benzene and tolusne.
b) Eapty tube, catalyst on wall; 0.028 in., thick wall, 1/4 in. 00.

Table 68 (Contd-4). MCH DEHTDROCENATION WITH VARIOUS CATALYSIS IN MICTR: RUNS 573-676

Ş	Catelyst	Catalyst			*	S MCH Correrston,	į	8	. 		
£	Ko.	Description	le ight	299	┢─	752			~	842	
626	10280-130	<pre>iube No. 1G, -35 Pt/405 type ! support (!) 405 type ! support (2) 205 type 6 binder Filled with quarts</pre>	0,089	28, 28, 27 65, 62, 62 88, 87, 87	8	55, 62	. 62	8	87,	25	
229	10.280-170	Tube Mc. 10, -3% Pt/40% type I support (1)*) 40% type I support (2) 20% type 6 binder Filled with quartz	0°08	16, 21, 22	8	50, 46, 46 61, 60, 60	9	9	8	23	
£28	9874-139	1\$ Pt/ UOP R-6 :yps AlgCs (ref)	0.424	22, 22, 23 47, 48, 48 68, 68, 70	8	17, 48	.	68	. 189	2	
<u>52</u>		:0220-:538 Binetailic, 10, 5%/type i support	628.0	25,(14),17 41, 37, 36 55, 56, 53	<u>~</u>	11, 37	38	55,	5 8	B	
93		10280-154A Bisetaliic, 9, 95/type I support	0.905	٧		13, 7, 6	9		17, 14, 17	13	
នី		10290-1548 Binstallic, 10, 5%/typs support	0.828	23, 18, 18 40, 36, 35 58, 54, 50	∞_	36	, 35	8,	35	8	
223	3551-3820i	10280-1550 5% metalb)/type support	0.798	19, 19, 17 (6), (4), (4) (14), (11), (9)c)	<u></u>	(6) (6) (6)) i	<u> </u>	5	=====================================	30
633		10280-1550 5% meteib)/type i support	0,336	24, 16, 15 41, 35, 34 (16), (14), (13)	17.	£1, 35	*	<u> </u>	<u> </u>	ت ئان	6.0
55	10280-170	10280-170 Tube No. 10, top 7-1/2" quartz filled	0,089	26, 23, 23 42, 47, 46 53, 52, 62	8	12, 47	. 68	53,	623	29	
# # -	safy tube, c.	 s) Expry tube, catalyst on mail; 0,028 in, thick mail; 1/4 in, 00, lapregnated in chelated force. 						5	1 T	(Sortinued)	İ

Expty tube, catalyst on mail; 0,028 in, thick wall, 1/4 in, 00.
Impregnated in chaisted form.
Bricketed numbers benzame, unbracketed numbers total conversion of MCH to benzame + tolumns

Table 68(Contd-5). HIS DESTINOURATION WITH VARIOUS CATALESTS IN SITES 573-675

چ	Catelyat	زهزه إغفز		•	S ICH Conversion,	بار ان بار
ŝ	0	Description	Teight	229	752	2118
33	10280-157A	Bimetallic, 1, 35/type I support	0.772	20, 22, 22	11, 13, 13	71, 68, 68
SS SS		10280-1579 Bimetallic, 3, 15/type I support	0.713	24, 23, 73	27, 44, 43	71, 68, 68
25		10280-1598 Binetallic, 5, 85/type I support	0.785	V	⊽	٧
83	1028G-155A	Simetallic, 5, 85/type I support	7,747	13, 11, 10	28, 25, 24	अर 'ऋ 'अ
633		10285-153C Trimetallic, I, 1,35/type I support	0.304	22, 25, 23	47, 46, 45	Ę
049		10280-161A Intestallic, to 1, 35/type I support	0.74!	17, 11, 17	42, 33, 33	23, 52, 53
541	10280-1518	10280-1518 Irimetmille, 1, 1, 3%/type 1 support	0.748	18, 17, 19	45, 40, 40	67, 66, 65
642	9874-139	15 Pt/USP R-8 type Algo, (ref)		•		
643		10280-1590 Irimetallic 1,5,8%/type 1 support	0.785	22, 19, 21	44, 42, 40	63, 61, 60
779	10280-151C	Triretailic, 1,1,35/type i support	0.750	21, 22, 22	19, 44, 44 69,	59, 67, 67
913	0191-0820:	Intactailic, i,i,3%/type I support	922.0	15, 18, 18	<u>;</u>	10,(49, 64, 64
949	3191-08201	Trimetallic, 1,1,3%/type I support	0.745	72, 19, 21	17, 41, 40 66,	66, 61, 50
239		10280-161F Trimetallic, i,1,35/type I support	0.753	20, 16, 18	38, 36, 35	38, 58, 58
848	10280-1785	35 Pt/435 type I support 435 type IS support 145 type 6 binder	0.478	25, 21, 22	53, 51, 51	83, 79, 74
649	10280-1788	Tube No. 13, -3% Pt, limed with above (7.75%)	0.327	31, 31, 32	73, 76, 76	35, 95, 96
930	10280-178C	Tube Ho. 14, -35 Pt. 11med with above (4.25")	191.0	31, 30, 31	18, n, n	96 '5: '96
65!	10280-170	Tube No. 10, Ested with 0.177" dia errula: tube of run 626	0.089	18, 17, 16	39, 40, 37 56, 55, 56	Se, 55, Se
652	652 9874-139	1\$ Pt/UOP R+3 type Al ₂ O ₃ (rat)	924°D	25, 25, 26	25, 25, 26, 52, 48, 47 75, 72,	15, 77, 71

(Continued)

Table 68(Contd-6). MCH DEHYDROGENATION WITH WARIOUS CATALYSTS IN MICTR: HUNS 573-676

S.	Catalyst	Catalyat			~	\$ ICH Conversion,	8	Ē	र्द्र	4	
2	No.	Bescription	[sight		253	}	22	~	<u></u>	æ	
653	8181-08201	Bimetallic, 10, 85/type 1 support	0.810	ě	16, 16, 17		£2, 38,	2 42	55	8	83
654		10280-1810 Trimstaille, 5,5,8%/type f support	0.750	≅.	17, 18	**	£3, £	3 3	65,	8	a
655		10280-1814 Trimetallic, 5,5,5%/type i support	0.764	5,	. , .	12 2	25, 7X	83, t6	R	5,	~
656		10280-1810 Trimetallic, 5,5,85/type i support	0.773	ž	14, 1	13 2	27, 22	22, 23	28,	Ξ.	on
657		10280-1634 Trimetallic, 1,3,1%/typs I support	0.m	ឌ	19, 2	22	46, 4	43, 44	77.	83	23
889		10280-1638 Trimetallic, 1,3,15/type I support	192.0	ୡ୕	20, 2	23 4	Ť,	45, 4!	71,	8	8
653		10280-1630 Trimetallic, 1,3,15/type support	111.0	24,	72, 72	24 54	8	47, 46	75,	, 7,	92
8	0291-08Z01	Trimetallic 1,3,1%/type 1 support	0.774	ដ	27, 26		47, 49,	3, 40	8	. 76.	36
199	10280-172	45 metal/type I support	0,740	55	z.	67	43, 4	41, 42	52,	57,	15
299		10286-1724 Bime:a:11c 4.45/type support	0,833	~	~	1	25, 27	n, n	£2;	×,	33
83	EZ21-08ZD1	Bimetailic 4,45/type i support	0.820	ដ	22	77.	46, 4	44, 43	159	5	8
999		10280-163A Trimetallic 1,1,3%/type I support	0,773	21,	24, 2	717	48, 4	44, 45	12,	હ	63
999		10280-163F Trimetallic 1,1,35/type i support	292.0	22	- -	``	£2; 34	38, 38	25	ង	52
999	8874-139	15 Pt/UOP R-6 type Aig0s (ref)	0,426	ឌ	23, 2	23	51, 49,	3, 47	<u>_</u>	, 72,	2
299		10280-185A 4\$ Pt/type 2 eupport	0,375	ខ្លុំ	2, 2	25 22	55, 40	19, 47	85	. 76,	, 76
999	8581-06201	Bisetallic, 3,1%/type 2 support	0,385	.55	22,	13	ន	47, 46	<u> </u>	, ×,	7.
269	3581-08201	Bimotallic, 2,25/type 2 support	0,398	26,	2,2	22	≆ Si	48, 45	2,72,	, 73,	1
දි		10280-1850 Binetaillic, 1,35/typa 2 support	0,333	ξ.	2, 2	72	13' 12	13, 42	2 67,	8	. 67
129	_	10280-18% 4% setal/type 2 support	0.400	23,	23, 15, 18		, ÷	17, 42, 41 58,	8	¥.	25

Table 68 (Contd-7). MCH DEHUROCEMATION WITH VARIOUS CATALISTS IN MICTR: RUNS 572-676

2	Cobalinat	Catalyst		* XCH	\$ MCH Conversion, T	
o	No. No.	Description	Colght CE2	233	352	842
229	10280-1788	672 10280-1788 Tube No. 13 rerun, cf 3an 549	0.377	32, 23, 23	0,327 32, 23, 23 80, 16, 73 96, 96, 96	36 36 36
673	10280-1780	673 10280-178C Tube No. 14, rerun, cf Run 650	19:00	3, 21, 23	23, 27, 23 72, 69, 63 92, 93, 97	£5 °E5 °78
929	10280-185F	674 10280-185F 4f mate18)/type 1 support	0.811	16, 9, 7	[16, 9, 7 21, 12, 10 29, 7, 9t)	29, 7, 95)
675	0290-130	675 10280-170 Tube No. 10, of Run (with quartz)	0,083	33, 26, 24	33, 26, 24 52, 48, 48 74, 72, 72	14, 72, 72
67F	ייייבניי-ניס	:Czew-i70 Tube No. 10, with O.;68º annular tube	n.089	10, 11, 12	U.089 10, 11, 12 33, 24, 26 31, 30, 22	31, 30, 73

a) impregnated in chelated force.
b) Some benzane formed with telume.

Table 69. MCH DEHYDROCFNATION WITH VARIOUS CATALYSTS IN MICTR: VARIO'S SUPPORTS

Partods June-August 1967

Conditions 10 sts pressure; catalyst reduced in H2 for 20 minutes at 796 %. BCH rate and cutalyst charge variable.

Run	NCH		Catalyet			Et of UCH
ko.	Rate, al/hm	No. 10200-	Description	Bounted on	Charging Teight, g	Converted to Toluene (752年)
			Grenular Catalyete	1		
311	90.)	63k	4% Pt/80% support type no. 1 - 20% binder type no. 6	granular	0.32	59, 58, 52
312	30	538	4% Pt/82% two supports type ro. ' = 20% binder type no. G	grenuler	0.44	58, 55, 54
338	90	75E	2% Pt/82% two supports type no. 1 - 20% blider type no. 6	grenular	0.51	52, 58, 55
		-	Catalyet Coatings			
320	45	64	# Pt/82% two supports type no. 1 - 18% binder type no. 6	tuba	0.26	29, 31, 31
321	45	G5A	4% Pt/82% two supports type no. (= 18% tinder type no. S	tube	lall	7
323	45	658	25 Pt/825 two supports type no. 1 - 185 binder type no. 6	tube	1,23	73, 79, 80
324	30	65B	2% Pt/82% tou supporte type no. 1 - 18% binos. type no. G	tube	1.23	45, 42, 39
326	45	6ôA	25 54/825 two supports type no. 1 — 185 binder type no. G	9CT 8417	ú .32	29, 40, 55
3?3	45	718	iž Pt/82; tuo supporte type no. i – 18; bindar type no. 5	tube	ე.3ე	38, 37, 3F
336	45	710	25. Pt/02% two supports type no. ! — 18% binder type no. 6	tube	0.34	56, 52, 53

A-HEPTANE DESTOROCTCLIZATION WITH WARLOWS CATALYSIS IN MICTE reble 70.

1.000

September-Movember, 1967
10 atm pressure, no added hydrogen. Catalysts reduced in hydrogen at 796°F.

Volume 0.9 ul catalyst diluted with 1.1 ml quarts chips.

IRSV = 10 (catalysts and quarts 13-20 mesh.) Conditions: Period:

Catalyst:

L		Catelynt		foreant	n-Kepta	ne Caner	forcent a-Neptane Comerted To Verfous Products	in a state of the	e te
<u> </u>	1		ng.		8427E			\$32.5	
	<u>.</u>	Descripti		Cracked Products	Penzene	Toluene	Crected Products	Perzen	Toluera
452	9874-139	15 Pt/R-8 type alumina (ref.)	я	-	-	6	,		
3 %	82.788	15 Ft/K-8 type stusing (refs)	% e		o 0	~ *	77-38	, ,	97-99
455	\$874-1038	Simetailie, 2, 2/4yps i support	2	7	0	-	9	6	•
8	10283-818	(% metal/type i support	5	`	-	78	` C	•	7
55 5	10280-638	42 metal/type i - type 6 support	2	~	0	2	2	==	#
3 5	28/4-1/0	• •	2	0	0	0	,	•	•
2 5	42/1-1/24	otel/type i	2	<u>~</u>	0	0	=	0	0
3 5	3874-121A	-	2	0	0	0	0	0	0
ğ 5		the metal/type 2 support	õ	2	0	9	=	•••	\$
79.	95-D9701	Sheralfype Z support	2	-	0	•	8	0	0
3 2	2000	•	2	=	ت	2	72	0	33
465	10230-14CB	Setal/type succeet	2 5	~ ~	=	o ,	•	9 (9 9
466	10280-744	Simetallic, 5, 44/ type I support	. 5	-	- C	5 C	• c	5 C	> C
(67	10250-868	15/ 1300 1	2	~	0		; ;	0	2
3 5	\$874-172A		0.	9	0	9	2.12	0	=
3 5	4020-1034	Binetallic, 5, 57/type I support	2	<u>ب</u>	0	~	••	0	0
? :		~*	2	=	0		5	0	2
	26/1-193A	Biretallic, 2, 5%type i support	2	····	0	2	2	o	15
=	initie, caspi	initiat complete cracking, than became completely inactive.						Confi und	(pen

Table 70 (Contd). n-HEPTANE DENYDROCYCLIZATION WITH VARIOUS CATALYSTS IN MICTR

		Catalyst		Percent	n-Kepta	re Correc	Percent a-Heptana Converted To Verlous Products	·loue Prod	uete
\$ 5			<u> </u>		3,2)2			\$,256	
	3	Bescription		Cracked Products	Berzens	Berzens Tolvene	Crecked Products	Benzene Tolucne	Toluene
111		10280-790 Stastalic, 1, 15/type I support	2	22		0	•	G	,
£73	_	Bisetellic, i, 15/type i support	2	~	ن		· =		• •
474	6749-338	Bimetellic, 33, 62% metal	9	•	•	•	87-74		
475	6246-119	Bisetallic,	9	2	0	9	9	-	0
476	99999	5646-64 Simetalife, 70, 30% metai	2	0	0	-	· ·	· C3	G
113	26-9759	Simtalife,	5	~	0	0	~		9
8:	6646-620	6645-630 ; Proprietury	ల	0	0	0	0		0
£79	10280-111A	20% metal/type I support	5	•	•	,	8	0	0
8	10780-1118	We matelftype I support	9	87	0	0	•	•	
£8	018-0920,	6 metal/type I support	9	Ħ	0	R	×	0	2
182	10280-1175	Cimetallic, 2, 2/type i support	9	8	•	0	•	•	•
8	811-08201	10280-118 Bisetailie, 2, 5.5%/type i support	::	•	0	0	•	0	۵

SD/M-T Coker Flushing Procedure

To essure adequate coker cleaning and avoid contamination from cold zone deposits of prior runs, a standardized solvent washing procedure has been adopted.

After the test fluid has been drained and the preheater-filter assembly removed, a 1/4" s.s. line is installed so that fluid circulation can be carried out. The water to the cooler is disconnected and replaced with a steam supply. This allows the system to be flushed with hot solvents. Further, the pump speed is increased to give several fold the normal 6 lb/hr flow. The following sequence of solvent flushes has been found satisfactory.

1st Flush:

Solvent by volume: 1/3 Dimethyl Formamida

1/3 Toluene 1/3 Acetone

Experience has shown that one 15-minute flush with this potent solvent mixture is sufficient to remove all deposits which might otherwise be dissolved by the warm hydrocarbon test fuel.

2nd Flush Series:

Solvent by volume: 1/3 Toluene

1/3 Acetone

1/3 Ethyl Acetate

A minimum of 2-15 minute flushes are made, or as many as required to produce a water-white effluent.

jrd Flush Series, Three Final 15 Minute Washes: Solvent by volume: Normal Heptane

Ecliesting the finel week the greater is executed to

Following the final wash, the system is opened to atmospheric pressure at various points and each segment dried with compressed nitrogen.

Finally, the test units are replaced, the system closed, and about 25 in. Hg vacuum imposed for at least 20 minutes. The test fuel is drawn in at the end of this period, with the vacuum still on. Besides drawing residual solvent out of the system, the vacuum reduces trapped pockets of air and so helps to equilibrate the fuel with the sparge gas which has the proper oxygen content.

Purification of RAF-161-60 Decalin

Decalin has a number of advantages as an endothermic fuel, mainly its lower vapor pressure and its better thermal stability (compared to MCH). In order to have an adequate supply of this material on hand, we obtained thirty drums from the Air Force fuel bank (RAF-161-60). This had been in storage since 1960. In spite of the fact that it was inhibited, and care taken in the selection of the material and the low temperature storage conditions, its color and thermal stability had deteriorated substantially.

Samples of this material were analyzed, purified and than evaluated in the bench-scale reactor by comparing the reactivity and stability for dehydrogenations with that of our F-113 Decalin. This work was undertaken to devise a means of purifying the 1500 gal of Decalin.

Analysis

The compositions of the various drums were determined in our laboratory by standard GLC techniques. Analyses were made of drums 1, 2, 16 17 and of mixtures from drums 3 to 15 and from 18 to 30 with the following results (Table 71).

Table 71. GLC ANALYSES OF RAF-161-60 DECALIN

Drum No.	trans-DHN, S	ois-DHN,	THN, %
1 2 5-15 mixture 16 17 18-30 mixture	33.2 33.2 33.7 33.1 34.0 33.8	66.0 66.0 65.5 66.1 65.1 65.3	0.8 0.8 0.8 0.9

On the basis of the above analyses it was concluded that the Decalin probably came from the same lot and that a purification procedure worked out for one of the drums would be suitable for the complete lot.

Sulfur content of the material as received was low and analysis of one drum showed 2.3 ppm S.

Purification and Evaluation

In previous work with Decalin the standard pretreatment was to pass the decalin through a 1-in, dismeter by 12-in, long silica gel column. A high purity Davison Grade 950, 60-200 mesh silica gel was used for this purpose. As this material was suitable for removing color, olefins and sulfur, it was of interest to see if a silica gel treatment would purify the 30 drums of decalin satisfactorily. For this purpose both Davison Grade 950 (high purity; 60-200 mesh) and Grade 28 (less pure; 20-40 mesh) were tested. The cost of the former was about 77.00/lb and that of Grade 28 was 54/lb. Both gels appeared to be equally effective in removing the color impurities (visual inspection), neither gel lowered the sulfur content below 2 ppm, and both gels improved the reactivity of the decalin and the stability of the catalyst markedly over the decalin as

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received (Table 72), when tested at 10 atm pressure, 1022°F, and IHSV of 100. For example, with the untreated decalin 39.50 conversion and a catalyst temperature increase of 254°F during the run was observed, while for decalin passed through a 12-in. silica gol column, 480 conversion and catalyst bed temperature increases of 38° to 47°F were observed. The increase in catalyst bed temperature during the run was taken as a measure of catalyst deactivation. In these tests the Grade 10 silica gol appeared just as effective as the more costly Grade 950. Thus, it appears that for catalytic dehydrogenation a silica gol treatment of the RAF-161-60 vill be satisfactory.

Tests were also done to see if the silicingel treatment would improve the thermal stability to an acceptable value, and also to determine how much silicated would be needed to purify the 50 drums of Decalin. The treatment consisted in passing DHN through a silicated column (2-in. diameter x 11-in. long, ca 500-600 ml volume) and collecting the effluent in 500-600 ml samples. Steam-jet gum, microgum and Erdeo Coker tests were then done on the samples. Because the DHN as received was colored, a light transmission measurement (Beckman DU Spectrophotometer) was carried out concurrently on the effluent to see if the DHN purification could be monitored by a light transmission technique.

Steam-jet gum, microgum, and spectrophotometric light transmission observations of successive samples showed that Grade 950 and Grade 28 silica gels were not depleted until just after 40 to 1 and 60 to 1 volumes of decaling per volume of silica gel had been treated, respectively. The depletion of silica gel activity was very sharp in each case and corresponded to the exact time when the visual color band reached the bottom of the silica gel column. No gradual change in purity of the effluent was observed in either case.

Table 75 shows a comparison of the effectiveness of the two grades of silica gel for material taken as near the silica gel depletion point as possible, considering the volume of sample required for a test. However, for the coker tests this amounted to about 5 gal, or about the last 20 volumes prior to the break point. A slightly better result was indicated by light transmission for the Grade 950 compared to the Grade 28 near the saturation point, but this is probably not significant. Grade 950 also looked a little better in the coker results, but, once more, the difference is so small as to be of doubtful significance. The most interesting result was that Grade 28 purified about 50% more Decalin than Grade 950, despite the coarse grain size of the former (20-40 mesh vs 60-200 mesh). Treating rates were kept about equal by use of a stopcock flow control on the Grade 28 column. A second important observation was the approximately 300°F improvement in thermal stability effected by silica gel treatment.

No eignificant differences were observed in gum contents. Interestingly, the Decalin had a rather marked yellow color prior to treatment. The colored material was found to be rather volatile so that most of it was vaporized in both gum tests. Hence, the effectiveness of the silica gel was not properly disclosed by the gum analyses. Attempts will be made to characterize the adsorbate since it appears to have such a deleterious effect on thermal stability.

Table 72. DERIDROGENATION OF RAF-161-60 DECALIN

Catalyst: 1, Pt on Algo, Feed: 33.2% trans-DHN Cetalyst Volume: 7 ml 56.0% cis-DHN Pressure: 0.8% THN LISY: 100

Run 160.	116	116-1	711	ਨੂੰ	135	136
Feed Pretreatment Silica Gel Column	none	- Grade	← Grade 950 →		Crade 28	
Temperature, "F Block Wall Catalyst Bed Profile	1022 076-955 506-955	351-265 351-79	102 — 102 — 51-76 141-79 141-202	왕 89-786 87-783	922 42-527 57-559	1022 015-22 678-736
Mary, Catalyst bed	42-127 52-57 54-67 42-57	27-27 27-28 28-28 28-28	25-057 85-057 72-057 74-057	635-40 640-44 653-60 15	671-73 630-34 698-704 1.5	8-11- 22-23 24-7-7- 25-23
old Conversion, 'A	35.5	46.3	6-24	31.5	40.2	9.94
Rate Constants Zero Order, atm, sec ⁻¹ First Order, sec ⁻¹	34°-41	5.00 0.672	5.14 0.699	2.96 0.355	3.54 0.503	4.83 0.648

Teble 73. COMPARATIVE EFFECTIVENESS OF DAVISON GRADES 950 AND 28 SILICA

GEL IN THE PURIFY ATION OF RA M-161-60 DECALIN

Analytical Test	RAF-161-60 "As Received"	RAF-161-60 Treated at 40:1 With Grade 950 SiO ₂	RAF-161-60 Treated at 60:1 With Grade 28 SiO ₂
Steam-jet Cum, b) mg/100 ml	0.6 ± 0.3	0.5 ± 0.3	0.0 ± 0.1 0.0
Microgum, c) mg carbon/100 ml	0.16 ± 0.02	0.07 ± 0.03	0.09 ± 0.02
Erdco Coker,d) 250 paig, °F 300 400 475 625	2/5 4/23.5 6/32.5	3/20	4/24
Light Transmission, 6) % (relative)	80.0	100.0	97.5

a) Grade 950 was high purity, 60-200 mesh; Grade 28 was lower purity, 20-40 mesh.

b) ASTM Method Designation: D381-61T (450°F, uteam, atmospheric pressure).

c) Method described in Technical Documentary Report No. APL TDR 64-100, Part II, pp. 180-187 (500°F, nitrogen, <2 mm Hg pressure).

d) Coker operating conditions: fuel flow, 5 lb/hr; preheater and filter

ot came temperature; air saturated fuel, but helium gas drive.

e) Beckman Model DU Spectrophotometer, 350 mm wavelength; 1 cm thick cells.

Purification of a 50-Drum Batch of Decalin

Following laboratory investigation of purification techniques we have purified the entire 30 drum batch. The quality of the product obtained was equal to that of the laboratory product.

The equipment for this process consisted of a stainless steel tank about 5 feet high and 18 inches in diameter, filled to a level of 5.3 feet with the Davison Grade 28 (20-40 mesh) silica gel. The column was prepared initially by pulling a vacuum of about 29" Hg on the silica gel with the vessel tightly closed. After several hours at this pressure, about 20 gals of laboratory purified Decalin was drawn into the column to vet the gel. The column was then filled with unpurified Decalin to within a couple of inches of the top.

During operation, Decalin was pressured into the top of the column from an original drum at the rate of about 55 gal per 7 hours, and was allowed to percolate down through the silica gal bed and flow into a fresh epoxy-lined drum (the first product was recycled). Flow was monitored with a needle valve and rotameter, and samples were taken at the beginning and end of each drum. In operation, a drum was put on stream in the morning and taken off at night, requiring little attention during the day since the pressuring gas (N₂) was automatically controlled. Liquid level in the column was maintained above the level of the gel bed at all times. After the samples had been checked spectrophotometrically by comparison with a "pure" reference sample, the drum was then pumped into a clean epoxy-lined 1500 gal tank. The total 30 drums were finally stirred thoroughly in the large tank by vigorous bubbling with nitrogen to assure constant composition during usage.

A Beckman Model DU Spectrophotometer, at 350 mu wavelength with 1 cm quartz cells, was used for the light transmission measurements. Light transmission was improved ca. 25 percent by the silica gel treatment, with no significant variations in the quality of samples during the 30 drum treating process. Thus, up to a volume ratio of about 39:1, Crade 28 silica gel performed its purification function without loss in effectiveness (150 lbs or 5.4 cu. ft of silica gel for 30 55-gal drums). Previous laboratory results showed the Grade 28 silica gel would purify RAF-161-60 without loss in effectiveness until a ratio of 60:1 was reached, after which purify declined rapidly. As can be seen, better than a 50 percent safety margin was allowed in the large scale treating cetup.

The 1500 gal tank is kept under nitrogen blanket at all times, and the Decalin has been mitrogen blanketed at all times since purification. Subsequently, 5 drums of this purified Decalin, now designated F-137, has been shipped to Pratt and Whitney Aircraft (Florida), 2 drums to Wright-Patterson AFB, and 1 gal to Edwards AFB. The balance is being used in our own heat transfer and thermal stability testing.

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Some of the adsorbate was removed from the column by flushing with heptane and acctone and has been recovered. Interestingly, it appears to be an effective vesicant on susceptible sking. A GLC trace of some of the material recovered in a laboratory run is shown in Figure 73. There is evidence for the presence of 5 major components. Because of the potentay of the total edsorbate as a thermal instability promoter it will be interesting to check the activity of the separate components after separation by GLC.



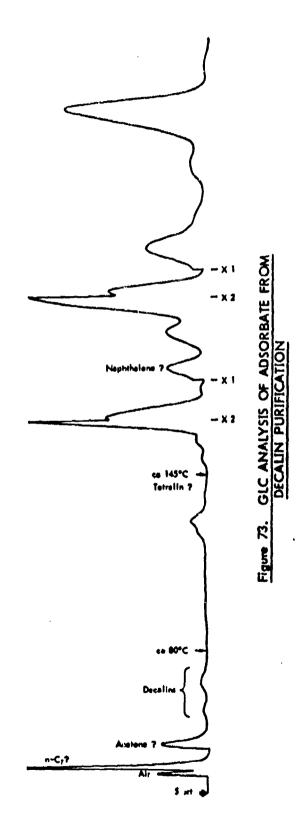


Table 74. FSSTR-HEAT TRANSFER TO HOR IN TROPIT 3/8" OD x

	22	EXI Feed Exte		1	Fluid Tamp, et Cauplings, T	4 t	Kent Finz b)	<u>;</u>	4	Total Bash
10018- -	19/4	(₈ H) (A)	ī	Section 1	=	ž	(kr) (fit)	Field	Outside Jailc)	Te Fiuid Btu/16
71-15:25	3	000'512	3		æ	=	2,78	# X :	15.	
				=	ë	ä	12,600	N R R R	3888	7 <u>19</u>
3.5:8	Ħ	244,000	ğ		8	210	98 5 *	8 2 5	និនិធិ	
				=	210	Ř	15,800	N N N	2 % 2 E	8 5
75-16:30	102	244,000	2	•	2	ş	15, 330	E E S	8 % 8	8
				=	g	ā	19,000	धे हैं हैं व	8 8 8 8 8 8	251
75-16:55	£03	241,000	875		B	Ë	21,300	<u> </u>	¥ \$ \$	<u> </u>
b) Heat	velocity.	Mess velocity. Hest flux based on inside tube surface.	tube su	rfscs.					18	(Continued)

b) Heat flux based on inside tube surface.
c) Tall thermocouples welded to side of tube.

Table 7s (Contd). FSSTR-HEAT TRANSFER TO MUH IN EMPTY 3/8" OD x 10 FT LONG SECTIONS: DAIA SUPMARY

3	ğ	BCE Feed flate		I _	First Temp. at Couplings, T	4 p. 4	Kest Flox b)	7 8 7.	14	1.4.1
1001 8-	16/hr	(hr) (H²)	a fa	Section	u)	ક	(F) (ff.)	Fluid	Outside Selic)	To Finid Rtu/Iis
				22	378	<u>S</u>	23,400	2 2 3	558 543 541	312
78-12:30	ĕ	241,000	2		*	9:0	16,800	H R R	8 # 8	ed
				=	310	£3	3,430	異異異	822	ā
3-i4:8	<u> </u>	241,000	8	8 00	Æ	ŝ	% 89,48	EEE.	\$ £ 8	æ
				=	3	5 8	25,530	£ £ £	65 SS SS SS SS SS SS SS SS SS SS SS SS SS	Ħ
78-15:40	101	241,000	Ř		2	Ġ	£.	E # #	888	£
			•	=	छ	8	25, 25	¥88	882	Ř

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b) Most flux turns on ensite tube surface.
 c) Well therecouples welded to side of tube.

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Table 75. FESTR: DATA SUMMARY SERIES 10013-90

Heat Transfer to MCH in Miniature Heat Transfer Section

Reactor No. 10018-82; 0.0265 in. ID x 0.018 in. wall x 6.0 in. long, Type 316 S.S. Feed: MCH, 34.2 lb/hr = 8.93 x 100 lb/(hr-ft²)

Run Ho.	Monaured Fower, Stu	Te	uid mp, F	Pres ps 1		Tubes) Length,	Vall Temp °Γ		Heatd) Flux, Rtu	Cumu- lativee) Heat,
	hæ	In	Out	În	Out	in.	Outsideb)	Insidec)	(hr·ft²) x 10°3	<u>Rtu</u> hr
90-15:51	450	68	99	895	718	0.25 1.35 2.45 3.55 4.65 5.75 (0-6)	125 125 151 134 159 146	115 115 121 124 129 136	128 128 128 129 129 129 (128)	18 100 101 263 345 427 445
90-14:10	1,220	66	142	895	750	0.25 1.35 2.45 3.75 4.65 5.75 (0-6)	208 209 21.7 224 258 252	182 185 191 198 212 227	350 350 351 352 355 357 (353)	51 273 496 720 944 1,171 1,223
90-14:28	2,950	65	240	895	735	0.25 1.35 2.45 3.55 4.65 5.75 (0-6)	386 369 382 392 414 440	328 311 324 354 356 382	870 865 869 874 881 890 (875)	126 677 1,229 1,785 2,540 2,900 5,055
99-14:45	5,480	65	359	3 95	733	0.25 1.35 2.45 3.55 4.65 5.75 (0-6)	597 555 555 574 603 637	499 457 457 476 505 539	1,625 1,604 1,604 1,612 1,626 1,641 (1,618)	235 1,261 2,285 3,305 4,330 5,370 5,610
90-15:07	8,640	65	499	895	723	0.25 1.35 2.45 3.55 4.65 5.75 (0-6)	795 741 759 765 790 852	647 594 592 619 644 687	2,612 2,576 2,574 2,595 2,611 2,640 (2,601)	377 2,030 3,670 5,310 6,960 8,603 9,020
90-15:28	11,870	65	600	895	685	0.25 1.35 2.45 3.55 4.65 5.75 (0-6)	929 877 877 904 964 1,111	745 691 691 719 782 932	3,463 3,415 3,415 3,440 3,494 3,630 (3,476)	2,690 4,960 7,040 9,250 11,510 12,060

T.C.'s spot welded to outside wall at indicated inches from inlet end.

Measured. All T.C. junctions on one side of horizontal tube.

Calculated.

d) Corrected for losser. () values are average for entire tube.
 e) Net heat to fauld up to indicated tube length.

Table 76. FOSTR: DATA SUMMARY SERIES 10018-94

Heat Transfer to MCH in Miniature Heat Transfer Section

Reactor No. 10018-82; 0.0265 in. ID x 0.018 in. wall x 6.0 in. long, Type 316 S.S. Feed: MCL, 45.3 lb/hr = 11.83 x 10^6 lb/(hr·ft²)

Run No. 10018-	Measured Power, htu	Te	uid mp,		es.,	Tubes) Length,	Well Temp		Heat Flux.d) Ptu (ar-fta)	Cumu- lativee) Heat, Btu
	hr	in	Out	In	Out	in.	Outsideb)	Invide ^a)	¥ 10-3	hr hr
94-9:56	5 ;	50	52	8%	585	0.25 1.35 2.45 3.55 4.65 5.75 (0-6)	118 119 125 130 138 145	104 105 111 116 124 151	180 181 181 181 182 182 (181)	26 141 255 371 486 601 623
94-9:42	1,490	49	125	886	595	0.25 1.35 2.45 3.55 4.65 5.75 (0-6)	200 205 215 222 258 251	169 174 182 191 207 220	425 426 427 429 432 454 (429)	61 552 602 874 1,149 1,424 1,487
94-9:55	3,240	49	156	826	607	0.25 1.35 2.45 3.55 4.65 5.75 (0-6)	547 546 558 371 400 420	262 281 293 306 335 355	(970) 958 963 981 989	159 748 1,558 1,972 2,591 3,717 5,564
94-10;10	5,390	48	280	886	598	0.25 1.35 2.45 3.55 4.65 5.75 (0-6)	504 492 506 526 566 595	404 392 406 427 468 497	1,581 1,577 1,582 1,591 1,608 1,602 (1,593)	228 1,232 2,256 3,244 4,260 5,288 5,525
94-10:19	7,660	48	356	886	590	0.25 1.35 2.45 3.55 4.65 5.75 (0-6)	641 621 635 658 707 742	507 487 501 524 575 611	2,245 2,255 2,241 2,257 2,287 2,306 (2,262)	324 1,749 3,170 4,600 6,050 7,500 7,600
94-10:27	10,210	48	457	886	573	0.25 1.25 2.45 3.55 4.65 5.75 (0-6)	764 743 760 783 826 878	597 575 595 617 661 714	2,943 2,925 2,920 2,557 2,992 3,035 (2,965)	465 2,292 4,160 6,050 7,920 9,850 10,280
94-10:56	15,720	48	559	886	550	0.25 1.35 2.45 3.55 4.65 5.75 (0-6)	917 892 904 936 967 1,078	705 679 691 724 755 871	5,975 5,948 5,960 5,996 4,026 4,147 (4,004)	574 3,095 5,610 8,140 10,690 13,280 15,900

T.C.'s spot welded to outside well at indicated inches from inlet end. Measured. All T.C. junctions on one side of horizontal tube. Calculated.

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d) Corrested for losses. () values are everage for entire tube.

e) Net heat to fluid up to indicated tube length.

Table 77. FESTR: DATA CHAMARY SERIES 10018-98

Heat Transfer to Nitrogen in Miniature Heat Transfer Section

Reactor No. 10018-97; 0.0265 in. ID x 0.018 in. wall x 6.0 in. long, Type 316 S.S. Feed: Nitrogen, 6.31 $1b/hr = 1.65 \times 10^6 \ lb/(hr \cdot ft^2)$

				Exp	erime	rital Data				Smoothe	d and Ca	lculated Data	
Run No. 10013-	Hea <i>s</i> ured		uid mp.	Pres		Tube Ve	11 Thermocou	ples		Vell T	çmp,	Heut Fluxd)	Cuera- lative*)
98-	Power, Btu/hr		c _	pe!		Loc	ation	Temp,	Longth, Inches		, 	Btu/(hrifts)	H-at,
	BCU/IIV	In	Out	In	Cut	lncheu ⁿ)	Positionb)	٠,	_	Outside	Inside	x 10 ⁻³	Btu/hr
14:19	195	68	160	Sills	h05	0.5 1.0 1.8 2.6 5.4 4.2 5.0 5.7	8 T 8 T 8 T	117 152 150 166 182 200 216 250	0 1 2 3 4 5 (0-6)	(111) 155 155 174 195 216 (257)	(108) 150 150 171 192 215 (254)	55.3 55.7 54.0 54.2	0 31 61 92 123 155 186
) by lak	66)	68	589	524	566	0.5 1.0 1.6 2.4 4.2 5.0	D T B T B	227 281 359 394 452 504 567 614	(0-6)	(210) 281 352 422 493 563 (634)	(197) 268 359 410 481 551 (622)	180 166 191 196 200 201 206 (195)	0 106 214 526 440 557 675
15:05	1,059	68	586	524	525	0.3 1.0 1.8 2.6 3.4 4.2 5.0 5.7	B 1 B T B T	526 420 508 595 690 778 880 962	0 1 2 3 4 5 6 (0-6)	(300) 481 533 650 765 881 (995)	(261) 400 515 655 748 864 (979)	284 295 502 510 518	0 169 327 499 676 857 1042
15:25	1,646	68	878	524	227	0.5 1.0 1.8 2.6 5.4 4.2 5.0 5.7	7 10 17 18 18 17	482 654 777 911 1,060 1,198 1,353 1,478	0 2 3 4 5 6 (0-6)	(455) 634 815 991 1,170 1,349 (1,528)	(429) 609 788 968 1,147 1,327 (1,506)	457 457 476 495 515	0 246 504 774 1,054 1,546 1,648

a) T.C.'s spot weided to outside wall at indicated inches from inlet end bus bar.
 b) Location of T.C. junction on horisontal tube. B = Bottom, T = Top.
 c) Outside wall temperatures by amouthing experimental data. Inside temperatures by calculation.
 d) Corrected for losses. Values in () are average over entire heated length.
 e) Net heat to fluid up to indicated tube length.

Table 78. FSSTR: DATA SUMMARY SERIES 10018-101

Heat Transfer to Vater in Miniature Heat Transfer Section

Reactor No. 10018-97; 0.0265 in. ID x 0.018 in. wall x 6.0 in. long, Type 316 S.S. Feed: Water, $54.1 \text{ lb/hr} = 14.13 \times 10^6 \text{ lb/(hr·ft²)}$

				Exper	iment	al Data				Smoothe	d and Co	lculated Date	
Pun No.	A neured		uid mp,		,	Tube We	11 Thermoco.	ples		Wei.1 1		Heat	Cumu- lative
101-	Power, Btu/hr		F	pe	14	Loc	etion	Temp,	Length, Inches	• 50	·)	Mura)	lative"
	beu/nr	In	Out	In	Out	Inches ^a)	Positionh)	• •		Outeise	Ineide	x 10""	Btu/hr
11:01	1,870	58	95	889	561	0.5 1.0 1.8 2.6 5.4 4.2 5.0 5.7	B T B T B	149 145 147 146 152 154 175 178	0 1 2 3 4 5 6 (0-6)	(148) 146 146 149 155 169 (199)	(107) 105 105 106 114 126 (152)	542 542 543 543 545 548 555 (544)	0 515 626 940 1,255 1,569 1,887
11:51	6,530	58	181	889	586	0.5 1.0 1.8 2.6 3.4 4.2 5.0 5.7	B T B T B	544 550 522 525 544 549 404 421	0 1 2 3 4 5 6 (0-6)	(34T) 352 321 350 355 397 (441)	(215) 200 189 198 225 265 (509)	1,906 1,872 1,882 1,890 1,714 1,953 1,995 (1,915)	0 1,098 2,188 3,279 4,379 5,500 6,650
12:05	10,180	58	254	989	579	0.5 1.0 1.8 2.6 5.4 4.2 5.0	B T B T B	459 459 450 485 485 576 602	0 1 2 3 4 5 6 (0–6)	(505) 471 455 465 502 569 (647)	(51%) 279 262 275 511 575 (461)	2,959 2,926 2,911 2,920 2,956 5,016 5,097 (2,960)	0 1,701 5,588 5,070 6,770 8,500 10,270
12:31	14,810	58	356	889	5175	0.3 1.0 1.8 2.6 5.4 4.2 5.0	# 1 1 1 1 1	647 606 597 588 633 648 689 716	0 1 2 3 4 5 (0–6)	651 613 591 611 645 607 (753)	387 347 325 345 380 423 (472)	h,540 h,290 h,260 h,290 h,540 h,590 h,260 (h,530)	0 2,496 4,970 7,440 9,950 12,450 15,010
19:01	21,040	58	b45	889	571	0.5 1.0 1.8 2.6 5.4 4.2 5.0	BTBTBT	746 729 752 753 812 824 900 927	0 1 2 3 4 5 6 (0-6)	(151) 151 141 119 821 888 (958)	(589) 389 400 434 484 548 (621)	5.950 5,850 5,850 5,920 6,010 6,070 6,250 (5,960)	0 3,579 6,760 10,170 15,620 17,110 20,670



a) T.C.'s spot veided to outside wall at indicated inches from inlet end bus bar.
b) Location of T.C. junction on horizontal tube. B = Bottom, T = Top.
c) Outside wall temperatures by smoothing experimental data. Inside temperatures by calculation.
d) Corrected for losses. Values in () are everage over entire heated length.
e) Net heat to fluid up to indicated tube length.

Table 79. ISSTR: DATA SUPMARY SERIES 10018-108

Heat Transfer To Water In Miniature Heat Transfer Section

Reactor No. 10018_103; 0.0265" ID x 0.018" wall x 6" long, Type 316 S.S. Feed: Water, 30.1 lb/hr = 7.86×10^6 lb/(hr·ft²)

			F	speri	menta	l Date				Fisio	othed an	d Calculated Date	1
Run No. 1001A 109	Manured Nover, I-lu		luia p, "F		nu., 1g		Vall Thermoe	ouples	Length, In.	Wall Te	_{ыр,} с)	Heatd) Flux,	Cumulative e) Heat,
10.6	7.0	In	Out	1n	Out	In. a)	Poritionb)	T p	111.	Cutside	Incide	(jm·142) x 10-3	br br
14:30	פדיד	હ્ય	91	111	1	0.5 1.0 1.8 2.6 5.4 6.2 5.0 5.T	B B B T B	100 107 106 112 113 120 120 125	0 1 2 3 4 5 6 (0-6)	(100) 105 109 115 117 121 (126)	(85) 86 92 96 100 104 (109)	218 219 219 219 220 220 221 (219)	0 126 272 379 306 633 761
14;40	1,209	62	105	107	1	0.5 1.0 1.8 2.6 5.4 4.2 5.0	B T B T B	122 152 151 160 160 160 150 150	0 1 2 3 4 5 6 (0.6)	(124) 129 155 140 146 151 (157)	(98) 105 109 114 120 125 (151)	557 558 559 340 341 342 343 (340)	0 195 590 586 785 980 1,178
26;47	1,967	62	129	104	1	0.5 1.0 1.8 2.6 3.4 5.7	B T B T B	155 172 171 182 181 197 199 210	0 1 2 3 4 5 (0_6)	(157) 166 175 184 195 202 (210)	(116) 125 154 145 152 161 (169)	54T 550 552 555 558 560 565 (555)	0 917 656 956 1,277 1,600 1,925
14:55	3,500	62	179	99	1	0.5 1.0 1.8 2.6 5.4 4.2 5.0	B T B T T B	219 245 241 260 260 285 305	(0.6)	(225) 255 24.1 260 274 209 (305)	(151) 161 175 186 200 215 (251)	1,008 1,013 1,019 1,026 1,035 1,041 1,050 (1,027)	584 1,171 1,162 2,556 2,956 3,560
15:02	b,150	62	200	1∞	1	0.3 1.0 1.8 2.6 5.4 4.2 5.0 5.7	B T D T B T	243 271 264 288 291 316 321 344	0 1 2 3 4 5 6 (0,-6)	(251) 260 274 290 307 526 (346)	(165) 174 188 204 221 240 (260)	1,186 1,191 1,200 1,209 1,220 1,231 1,245 (1,211)	0 687 1,578 2,074 2,777 5,490 4,200
15:15	5,370	65	256	106	6	0.5 1.0 1.8 2.6 3.4 4.2 5.0	B T D T B	288 315 315 359 364 374 385 415	0 1 2 5 4 5 6 (0.6)	(295) 306 321 341 364 390 (417)	(190) 201 216 256 259 205 (512)	1,497 1,505 1,516 1,551 1,548 1,568 1,568 (1,555)	0 868 1,761 2,622 3,510 6,610 5,520

a) T.C.'s spot welded to outside wall at indicated inches from inlet end bus bar.
b) Location of T.C. junction on horizontal tube. B = Bottom, T = Top.
c) Outside well temps by smoothing experimental data. Inside temps by calculation.
d) Corrected for losses. Yelues in () are average over entire heated length.
c) Met heat up to indicated tube length.

Table 80. FSSTR: DATA SUMMARY SERIES 10018-116

Heat Transfer To Water In Miriat re Heat Transfer Section

Reactor No. 10018_110; 0.0265" ID x 0.018" wall x 6" long Type 516 S.S. Feed: Water, 5.33 lb/hr = 1.39 x 10^6 lb/(hr·ft²)

	T	Ī		Exper	inen:	al Drie				Sa	onthed a	and Celculated Sut	
Run No. 10018. 116.	Mesured Power,		Luid P. *F		ess.,	 	Vall Thermon		Length,	Υ	emp, a)	Heatd)	Cumulative*)
	htu hr	:r	Out	În	Cut	:n.ej	Position ^b	Temp,	In.	Outalde	Inside	(hr-ft*) x 10-3	Prop her
10:58	260	250	225	50	ig.	0.5 1.0 1.8 2.6 5.4 4.2 5.0 5.7	B T B T B	245 256 265 265 265 265 263 260	0 1 2 5 6 (0•6)	(240) 255 265 265 264 262 (259)	(255) 250 260 260 259 257 (254)	76.8 " " (76.8)	0 k4 69 155 176 2.2 2.5
11:32	557	285	230	מ	6	C.3 1.0 1.8 2.6 3.4 4.2 5.7	BT BT BT BT	512 527 514 539 558 554 527 521	0 1 2 5 6 (0_6)	(509) 526 536 539 535 528 (518)	(298) 315 325 528 524 517 (507)	156	0 90 180 271 361 451 541
11:59	956	3 ∞0	:42	144	10	0.5 1.0 1.8 2.6 5.4 4.2 5.0 5.7	B T B T	395 395 399 389 384 380 373 367	0 -1 22 31 4 5 6 6) 0 -6 6)	(595) 595 591 586 581 575 (565)	(5Tr) 5TS 5TS 568 562 555 (54T)	265 (265)	0 155 306 459 612 765 918
12:24	1,595	563	248	172	12	0.5 1.0 1.6 2.6 5.4 5.7	9 1 8 1 9 1 8	417 414 410 405 405 405	0 1 2 3 4 5 6 (0_6)	(\$17) \$15 \$12 \$09 \$06 \$02 (599)	(592) 590 587 581 577 (574)	389 (389)	0 225 450 575 900 1,165 1,350
12:45	1,892	354	251	196	15	0.5 1.0 1.8 2.6 5.4 5.7	B T B T B	435 436 440 442 442 442 456	0 1 2 3 5 6 (0-6)	(435) 437 440 442 443 441 (436)	(597) 401 404 406 407 405 (400)	552 (552)	0 519 658 957 1,276 1,594 1,915
15:15	2,394	367		247	16	0.5 1.0 1.8 2.6 5.4 4.2 5.0 5.7	B T B T B T	464 478 473 481 480 480 475	0 1 2 5 4 7 6 (06)	(459) 474 479 480 480 479 (475)	(\$16) \$31 \$36 \$37 \$37 \$36 \$36 (\$32)	670 - (670)	0 387 715 1,162 1,549 1,936 2,354

a) T.C.'s mpot weided to outside wall at indicated inches from inlet end bus tir.
b) Location of T.C. junction on horizontal tube. B = Rottom, T = Top.
c) Outside wall temps by smoothing experimental data. Inside temps by calculation.
d) Corrected for losses. Values in () are average over entire heatel length.
e) Met heat to fluid up to indicated tube length.

Table 81. FSSTR: DATA SUMMARY SERIES 10018-119

Heat Transfer To Water In Miniacure Heat Transfer Section

Penotor No. 10018_110; 0.0265" ID x 0.018" wall x 6" long, Type 316 S.S. Feed: Water, 4.97 lb/hr = 1.30×10^6 lb/(hr·ft²)

			ř	uperi	.sent s	l Cata				Se	ooth and	Calculated Data	
Pur No.	Hessured	72	uld	Pre		Tube	Vall Thermor	ouples		V-2.1 T	emp, o)	Heatd)	Cumulative ^{e)}
10016	Power,	Tenj	, °F	pe	ig		wetion	Tomp,	Length, In.	•,	, · · ·	Flux,	Hemt, htu hr
	hir	În	Out	In	Out	in.a)	Fosttionb)	Y		Outside	Inside	(hr:(t*) x 10-3	hr
15:20	209	675	581	457	31	0.3 1.0 1.8 2.6 3.4 4.2 5.0	0 T B T P	446 445 437 438 430 430	0 1 2 3 4 5	(449) 444 439 434 429 444 (455)	(445) 400 435 430 425 440 (449)	59.0 59.0 58.8 56.8 58.9 59.0 58.9	0 94 68 102 126 170 204
15:44	49 5	478	368	\$77	34	5.7 0.5 1.0 1.8 2.6 5.4 4.2 5.0 5.7	B T B T B	451 458 453 449 495 510 530 544 562	(0.6) 0 1 2 3 4 5 6 (0.6)	(460) 454 448 502 525 545 (567)	(452) 446 440 494 515 537 (559)	(58.9) 134 134 134 136 136 137 137 (155)	0 78 155 255 512 390 470
15:56	6 k 2	476	419	490	35	0.5 1.8 2.6 5.4 5.0 5.7	B T B T B	462 460 460 538 557 585 605 630	0 1 2 3 4 5 6 (0_6)	(462) 461 460 547 577 607 (637)	(451) 450 449 537 567 597 (627)	171 171 173 175 176 177 178 (1%)	0 99 198 298 399 501 6CL
16:16	1,062	482	550	516	39	0.5 1.0 1.8 2.6 5.4 5.0 5.7	B T B T B	478 474 598 642 624 732 771 817	0 1 2 3 4 3 (0_6)	(480) 607 662 719 774 (850)	(465) 457 590 646 703 758 (814)	275 274 263 289 291 295 296 (286)	0 159 520 484 652 821 9 92
16:28	1,462	486	666	540	45	0.5 1.0 1.8 2.6 5.4 4.2 5.0 5.7	8 1 1 1 1 1	491 555 684 749 811 877 940	(0-6)	(555) 555 698 778 858 958 (1,018)	(552) 532 676 756 836 917 (997)	580 580 595 405 410 418 425 (401)	0 220 444 674 909 1,150 1,590
16:50	1,909	¥85	800	557	46	0.3 1.0 1.8 2.6 3.4 4.2 5.0	в в т в т	504 687 792 877 958 1,043 1,128 1,207	(0-6)	(687) 687 808 914 1,020 1,128 (1,254)	(658) 658 780 886 995 1,102 (1,208)	504 504 520 523 546 560 573 (552)	0 291 587 688 1,197 1,517 1,644

T.C.'s spot welded to outside well at indicated inches from inlet end bus bar. Location of T.C. junction on horisontal tube. B = Bottos, T = Top. Outside well temps by escothing experimental data. Inside temps by calculation. Corrected for losses. Values in () are average over entire heated length. Het heat to fluid up to indicated tube length.

Table 82. FSSTR: DATA SUMMARY SERIES 10018-126

Heat Transfer To Water In Miniature Heat Transfer Section

Reactor No. 10018_122; 0.0265" ID x 0.018" wall x 4" long, Type 316 S.S. Feed: Water, 99.4 lb/hr = 26.0 x 10^6 lb/(hr·ft²)

				aper	mente	1 Data				Sec	othed w	d Calculated Date	
Run No. 10018-	Measured Power,		luid p, "r	1	se.,		Wall Thermo	Couples	Length, In.	Voll Te	up,c)	Heat ^d) Flux, Btu	Cumpletive ^{e)} Heet, Blu
126-	jitu hir	tn	Out	In	Out	In. "	Position ^{b)}	remp,	10.	Outside	Inside	(hr·fts) x 10-3	hy:
13:40	2,360	67	92	981	204	0.25 0.75 1.25 1.75 2.25 2.75 3.25 3.50	8 T B T B	174 178 181 131 185 187 190	0 1 2 3 4 (0.4)	(169) 179 185 188 (193)	(92) 102 106 111 (116)	1,050 1,032 1,034 (1,052)	0 594 1,191 1,788 2,586
15:48	6,030	67	129	981	229	0.25 0.75 1.25 1.75 2.25 2.75 3.25 5.50	B T B T B	320 325 335 330 342 343 353 353	0 1 2 5 4 (0.b)	(305) 530 536 548 (364)	(120) 1 7 155 165 (181)	2,600 2,610 2,620 (2,610)	0 1,496 3,000 4,510 6,030
15:56	9,900	67	168	981	545	0.25 0.75 1.25 1.75 2.25 2.75 3.25 3.50	B T B T B T	462 465 485 475 499 57.1 511	0 1 2 3 4 (0.4)	(452) 476 484 504 (532)	(119) 194 202 222 (250)	4,260 4,280 4,320 (4,280)	0 8,550 6,920 7,600 9,900
14:04	15,960	67	226	981	248	0.25 0.75 0.75 0.75 0.75 0.75 0.75 0.75 0.7	B T T T B T	655 675 666 690 689 720 721	(OT)	(608) 664 679 704 (158)	(196) 254 270 297 (352)	6,630 6,670 6,720 (6,670)	3,820 7,660 11,530 15,420
14:10	17,590	67	244	981	249	0.25 0.75 1.25 1.75 2.25 2.75 5.25 5.50	B T B T B	697 706 727 719 747 746 782 783	(0.4)	(666) 715 732 765 (816)	(2:1) 262 280 315 (367)	7,860 7,500 7,580 (7,510)	0 8,290 8,620 12,970 17,360
16:25	19,440	67	265	981	256	0.25 0.75 1.25 1.75 2.25 2.75 5.25 3.50	B T B T B	164 110 191 192 820 820 860 860	(04) 2 8 9	(757) 783 807 841 (900)	(257) 286 511 348 (408)	8,380 8,440 8,530 (8,440)	0 k,820 9,690 14,590 19,540

T.C.'s spot welded to outside wall at indicated inches from inlet end bus bar. Location of Y.C. junction on horizontal tube. B = Bo.tom, T = Top. Outside wall temps by smoothing experimental data. Inside temps by calculation. Corrected for losses. Values in () are average over entire heated length. Net heat to fluid up to indicated tube length.

Table 83. FSSIR: DATA SUMMARY SERIES 10018-127

Heat Transfer To MCH In Miniature Heat Transfer Section

Reactor No. 10018-110; 0.0265" ID x 0.018" wall x 6" long, Type 316 S.S. Feed: MCH, 31.4 lb/hr = 8.20×10^6 lb/(hr-ft²)

			7	a po r	watu	l lute)	(Imc	uthed as	d Calculated Data	
Run No:	Measured	,	luid	Pre	50.	Tube	Well Thermoo	ouples		Vell Te	e,e)	Heat (1)	Cumulative*)
127-	Pover,	700	p, 'F	Pe	ig .	L.	ocation	femp,	longth, In-	.,		nu,	Heat, juy hr
ا	Nr .	In	Out	1n	Out	in. a)	(Valiton ^{b)}	* p		Outside	Ina lite	(hir-fit*) x 10-3	hr
10:45	1,054	67	180	9415	844	0.5	h	265	0	(;+4)	(250)	513	0
	}		ļ	i	1	1.0	Ţ	276	1	1717	-41	516	198
	1	,	ł	1	l	1.6	l B	391	?	1119	1 22	518	596 896
	i	1	l	l	Į į	2.6		294 304	}	500	; 464s	530 525	1,196
	ł	1.	l	!	i	3.2	P) T			511	:175	526	
	1	1	ŀ	l	l	5.0		305 302	1 6	\$350	(500)	5:5	1,500
		1	i	Į	•				(0.6)	(556)	()00/	(521)	1,001
	1					5.1	' '	350	(0.0)	i	i	(581)	
11:05	3,580	60	278	OFA	848	0.3	В	451	ها	(\$21)	(555)	1,007	1 0
,	,,,,,,		.,.			1.6	Ť	455	ĭ	496	370	1.01	1,8%
			ļ	ł		1.6	'n	452	ءَ	651	586	1,017	1.170
						8.6	T	456	,	166	l koï	1,022	1,710
					Ì.	5.3	3	475	(461	416	1,027	2,350
						4.2	۲	469	5	496	451	1,052	2,9%
		1				5.0	•	496	6	(511)	(446)	1.057	5,40
		1				5.7	T	506	(0.6)		1	(1,0%)	1
11,80	7,020	60	445	985	841	0.5	8	700	ა	(742)	(6:7)	1,997	0
	·			. 1		1.0	۲	658	1	165	Shill	1,976	1,148
						1.6	B	691	5	697	571	1,990	2,500
					1 1	2.6	Ţ	697		709	595	2,005	5,450
					i i	3.4		721		751	616	2,016	4,610
						5.8	Ţ	725	3	, נכד,	638	2,049	5,760
		1				5.0		756	6	(מזי)	((46)	0.013	6,960
			1			5.1	Ť	767	(0-6)	}		(2,006)	
11:40	10,510	60	584	986	821	0.3	B .	684	0	(935)	(772)	5,000	
1	i i	1			l 1	1.0	Ţ	0,6	1	846	660	2,990	1,757
						1.6 2.6	B T	873 916	₽ 3	671	707	5,010	3,470
					1	1.4	, i	916	2	951	755	3,050	5,350
						4.2		929	,	975	813	5,060 5,100	6,980 8,710
						5.0		981	6	(1,012)	885	5,160	
			1	i		5.7	Ť	1,014	(0.6)	(*,01.7	CII	(3, 248)	10,570
18:05	1,715	60	178	965	SLA.	0.3	,	29.4	0	(255)	(2.9)	472	٥
	i *''*/		- '-	~~		ĭ.ó	ï	165	i	100	216	\$75	:-/19
1			1			1.0	i i 1	286	è	278	244	478	549
			· .			2.6	i i	205	5	191	- 7	48)	827
- 1	i					5.4	i i f	293	` [303	269	484	1,105
1						6.2	Ť	799	. 3	516	THE .	487	1,587
1						5.0	, i	317	6	(529)	(194)	490	1,669
				i		9.7	T	325	(0-6)	''' '		(481)	•••

a) T.C.'s spot wolded to outside well at indicated inches from inlet six bus bar.
b) Location of T.C. junction on horisontal tube. B = Rottom, T - Top.
c) Outside well temps by emouthing experimental data. Inside temps by naioulation,
d) Corrected for lowess. Values in () are average over entire heated length.
c) Not heat to fluid up to indicated tube length.

Table 84. FIGTR: DATA SUMMARY SERIES 10018_129

Heat Transfer to MCH In Miniature Heat Transfer Section

Reactor No. 10018-110; 0.0265" ID x 0.018" wall x 6" long, Type 316 S.S. Feed: MCH, 76.5 lb/hr = 19.98 x 10^6 lb/(hr·ft²)

			}	15+1	re-tri s	Limin				(lee	othed an	1 Calculated Date)
Hun No. 1001 N.	Heamired Nover	, FI	 tuld p. "t		on.,	}	Vall Thermos	ongdø a	Longth,	Well Te	mp, a}	Heatd) Flux	Cumulative ^{e)} Heat,
1:9-	1111		····	ļ		<u> </u>	mation	Temp,	In.		,		Fits
	11.	j n	(h)t	10	Out	10.4)	institunt)	• 4		Ostalda	Inelde	the teal in rosa	hr
11:05	5,100	10	:134	1,3191.3	#	0.5	β T	5%1 550	0	(55e) 558	(131) 200	1,444	0
	1	l	l	į	(1.6	μ	319	•	576	179	1,476	1,649
	Į	1 .	ļ	1	j	2.6	1	385	,	397	117	1,490	2,550
	1	1	l	l i	ł	3,4	H T	410	5	413	314	1,505	5,410 4,199
	ł	i i	ł		1	3.0	'n	407	l 6	(452)	(333)	1,516	5,150
	•		Ī		İ	5.7	Ť	119	(0.6)		\ <i>''''</i>	(1,486)	1
11;.0	9,650	w	105	Mu	190	0.5	ıı T	336		(535)	(342)	2,710	0
	ł	ł	ì		ł	1.8	è	580	1 2) 5k5	375	2,790 2,760	3,100
	i			1	ľ	2.6	Ť	582	,	í án	130	2,750	4, 100
	{		1		1	9.4	н	ເກ	,	6:0	450	2,800	6,570
	i	1	!			4.1	1	615	3	655	ring plan	r Me	7,190
	Ì	l	l			5.0	† †	637	(O-6)	((A1)	(472)	(2,170)	9,6,0
11:40	10,4.0	59	31%	GH9	195	0.5	н	565	۰	(%1)	(557)	2,910	0
	į	1	1	}		1.0	T	>6%	1	575	392	2,940	1,692
	! ,	} {	1			1.8	h T	613	3	654	455	2,970 5,000	5,130
	ļ .	1 1				3.4	19	655	4	658	177	3.00	6,410
	f i	1 1		i I		4.1	T	649	9	616	496	5,010	H, 640
:						5.7	, p	682 680	(0-6)	(6/15)	(505)	3,050 (2,990)	10,560
		.,,		ren.	a	*		596	(55.1)	(,,,,,	,	}	
11:51	11,030	"	35%		1.4	0.5	h T	596	ĭ	(572) 604	(519) 413	5,100 5,150	1,800
		1		1]		1.6	B	(40		637	446	5,140	5,630
	· :					2.6	T	644	•	666	476	5,190	5,4%
				1 1		5.4	<u> </u>	691	•	691	501	5,0.0	7,110
	1			1 1		5.0	T	685 717	}	710 (7:1)	584 (559)	5,130 5,250	9,170
						5.7	ř	ฑ์เ	(2.6)	(1.4)	11504	(5,180)	11,040
12,05	1, (4k)	749	:06	721 4	199	0.5	. H	548	0	(335)	(255)	1,489	a
						1.0	, ,	91,4	1	359	257	1,507	HV4
						2.6	, i	585 589	2	381 402	2479 300	1,5,52 1,557	1,761 2,630
						3.4	i i	413	3	418	316	1.39	3, 4.0
	ĺ					4,5	1	410	5	430	550	1,559	4,4.40
		}				1.6	<u> </u>	455	6	(442)	(340)	1, 4/	3, 5.0
		L i		}		<u>5.7</u> j	Ť	497	(0.6;		L l	(1,554)	L

a) This again webled to detaile well at Indicated inches from inlet end bum bar.
b) Invention of let, jugetion on burizontal tube. He inition, T = Top.
c) this ide well temps by anothing experimental data. Inside temps by calculation.
d) Corrected for lusses. Values in () are average over entire heated length.
e) Ref leaf to finite up to indicated tube length.

Table 85. FSSTR: DATA SUMMARY SERIES 10018_130

Heat Transfer to MCH In Miniature Heat Transfer Section

Reactor No. 10018_210; 0.0265" ID x 0.018" wall x 6" long, Type 316 S.S. Feed: MCH, 58.3 lb/hr = 5.23×10^6 lb/(hr-ft²)

			ı	aperl	menta	1 Duta				Sec	othed er	d Calculated Date) }
Rum No. 10018. 130.	Heasured Power, Ptu		luid 9, °¤		ig		Vall Thereon	Temp,	Length,	Vell te	mp, °)	Heatd) Flux, htu 10-3	Cumulative ^{e)} Heat, Btu
	P. P.	ln	Out	In	Out	ln.a)	Positionh)	F		(citalde	Inside	(hr-ft2) x 10-9	hr
15:06	1,806	ધ	1,50	991	517	0.5 1.6 2.6 5.4 5.7	5 7 8 7 8	190 200 210 216 228 227 239 261	0 1 2 3 5 6 (0-6)	(18-) 149 211 221 230 239 (245)	(150) 162 175 18h ±95 202 -98)	506 509 512 513 517 517 519 521 (514)	0 274 588 686 1,184 1,483 1,783
13:21	5,580	62	190	991	526	0.5 1.0 1.8 2.6 5.4 4.2 5.0	1	300 308 332 337 355 354 372 379	0 1 2 5 6 (0-6)	(268) 512 531 546 559 571 (581)	(218) 242 261 276 209 301 (511)	998 1,009 1,019 1,026 1,032 1,038 1,043 (1,024)	580 1,166 1,757 2,551 2,950 5,550
13:52	7,190	43	300	991	519	0.5 1.0 1.8 2.6 5.4 4.2 5.0	8 T 8 T 9 T	498 500 540 546 578 574 604 611	0 1 2 3 4 5 (0.6)	(678) 508 536 562 584 603 (618)	(348) 379 407 434 456 476 (491)	2,017 2,057 2,056 2,075 2,087 2,099 2,109 (2,069)	0 1,172 2,555 5,550 4,750 4,960 7,180
23:44	10,490	63	386	991	502	0.5 1.0 1.6 2.4 7.0 5.7	B T B T B	इ.स.च.च.५५ इ.स.च.च.५५५	0 1 2 3 4 5 (0.6)	(626) 654 687 717 743 760 (773)	(199) 177 511 512 569 586 (599)	2,950 2,950 2,960 3,010 3,050 3,050 3,060 (3,000)	0 1,699 5,410 5,150 6,890 8,650 10,410
24109	16,130	64	676	991	472	0.5 1.6 1.6 2.6 5.4 5.0 5.7	8 T 8 T 8 T	790 777 854 855 865 874 912 916	0 1 2 3 4 3 6 (0.6)	(768) 795 828 858 859 909 (926)	(541) 569 635 64 686 (20)	3,960 3,990 4,030 4,070 4,100 4,130 4,150 (4,060)	0 2,298 4,620 6,960 9,520 11,700 14,090
14:11	15,630	65	509	991	457	0.5 1.0 1.8 2.6 5.4 4.2 5.0 5.7	B T B T B	850 835 895 890 945 954 980 993	0 1 2 3 4 5 6 (0.6)	(327) 851 834 916 947 976 (1,005)	(581) 606 640 675 705 775 (765)	h, 370 h, 650 h, 650 h, 550 h, 570 h, 600 (h, 690)	0 2,537 5,100 7,680 10,280 12,970 15,560

T.C.'s spot welded to outsice well at indicated inches from inlet end tus bar.
Location of T.C. junction on horisontal tube. B = Bottom, T = Top.
Outside well temps by smoothing experimental data. Inside temps by Jacoulation.
Corrected for losses. Values in () are average over entire heated length.
Met heat to fluid up to indicated tube length.

Table 86. PSSTR-DATA SUPPURT SERIES 10018-131: HEAT TRANSFER TO MCH IN MINIATURE HEAT TRANSFER SECTION

Reactor No. 10018-110; 0.0265" ID X 0.018" Well X 6" Long Type 316 S.S. Feed: MCH, 5.95 1b/hr = 1.56 x 10° 1b/hr·ft²)

Fancth, Fast, Fa			}	(T DOCK	and a	Experimental Data				Secot	ped and	Secothed and Calculated Data	,
Post Post	F. F.		ĮŠ.	胃	Ē		Tube V	all Thermocon	uples		Tal.	3)	Heat d)	Cummistive"
The continue of the continue	1001	Power, Btu	H	· .	200		3	cation	ř.	नुष्टी सुर्वे	•		Bin 210-1	Bet.
392 67 173 697 838 0.3 E 233 0 (165) (158) 1.6 1.7 T 505 3 450 443 2.6 T 505 3 535 529 2.6 T 606 604 604 4.2 T 608 637 6 604 5.0 B 637 6 631 604 6.7 T 608 (3-6) (579) 1.0 T 465 1 465 455 1.0 T 465 1 465 455 1.0 T 465 1 465 455 1.0 T 608 (3-6) (340) 1.0 T 608 608 601 2.6 T 650 601 601 4.2 T B 500 501 4.2 T 608 60-6 601 5.7 T 645 606 601 6.0 800 830 6 606 601 6.0 800 830 6 606 606 <td< th=""><th></th><th>ä</th><th>ā</th><th>Out</th><th>អ</th><th>Out</th><th>In. a.</th><th>Position^{b)}</th><th>•</th><th></th><th>Outside</th><th>Inside</th><th>(Br.rt.)</th><th></th></td<>		ä	ā	Out	អ	Out	In. a.	Position ^{b)}	•		Outside	Inside	(Br.rt.)	
562 68 228 897 899 0.3 B 637 6 665 631 655 631 655 631 655 631 655 631 651 652 632 632 632 632 632 632 632 632 632 63	14:03	392	67	173	897	838	0.3	323	233	0	(165)	(158)	85	٥
582 68 228 897 898 0.3 B 401 2 450 445 582 68 228 897 898 0.3 B 603 604 1.0 F 608 (555) (579) 1.0 F 608 682 3 725 60 5.0 B 5.0 B 601 601 5.0 B 602 603 603 5.0 F 603 603 604 5.0 F 603 605 5.0 F		}	;					6-	35	М	345	338	102	8
562 68 228 897 898 0.3 B 637 6 (555) (579) 562 68 228 897 899 0.3 B 637 6 (555) (579) 562 68 228 897 899 0.3 B 570 0 (250) (240) 1.6 B 587 2 610 601 2.6 T 682 3 725 716 3.4 B 580 6 (355) (375) 5.7 T 7 T 416 (0-6) (355) 5.8 5.0 B 520 6 (355) (375) 5.8 5.0 B 520 6 (355) (375) 5.9 5.0 B 520 6 (355) (375) 5.0 B 520 6 (355) (375) 5.0 B 520 6 (355) (375) 5.0 B 521 2 860 847 5.0 B 521 2 860 847 5.0 B 521 2 860 847 5.0 B 521 2 860 847 5.0 B 521 2 860 847 5.0 B 521 2 860 847 5.0 B 521 2 860 847 5.0 B 521 2 861 5.0 B 521 2 861 5.0 B 521 2 861 5.0 B 521 2 861 5.0 B 521 2 861 5.0 B 521 2 861 5.0 B 521 2 861 5.0 B 521 2 861 5.0 B 521 2 861 5.0 B 521 2 861 5.0 B 521 2 861 5.0 B 521 2 861 5.0 B 521 2 861							1.8	Д	÷.	K	654	3	105	'n
562 68 228 897 898 0.3 B 570 4 610 604 562 68 228 897 898 0.3 B 310 0 (250) (240) 1.6 B 587 2 610 601 2.6 F 682 3 725 10 601 3.4 B 580 682 3 725 116 5.7 F 682 3 725 116 5.8 F 682 3 725 116 5.0 B 5.0 B 580 890 890 890 890 890 890 890 890 890 8							% 9.0	H	202	n	535	223	106	178
5.0 B 637 631 5.0 B 637 637 631 5.0 B 637 638 (578) 5.0 B 10 0 (558) (578) 1.0 F 608 0 200 (540) 1.0 F 608 1 455 455 1.0 F 682 3 725 716 2.6 F 830 5 610 601 4.2 F 830 5 560 551 5.0 F 430 0 645 335 5.7 F 416 0 645 635 1.0 F 647 1 645 631 1.0 F F 847 3 770 757 2.6 F F F 847 3 770 757 5.0 F F F 530 6 6513 6514 6.7 F F F 7 7 7 7 7 F F F 7 7 7 7 7 8.4 F							4.5	A	570	*	019	3	101	239
5.0 B 637 6 (555) (579) 5.7 T 608 (3-6) (240) 1.0 T 465 1 455 455 1.0 T T 455 116 601 2.0 T T 455 116 601 4.2 T T 430 5 560 551 5.0 B 550 5 551 551 65.7 T T 416 0.46 1 645 631 1.0 T T T 446 1 645 631 1.0 T T T 645 631 1 1.0 T T T 645 631 1.0 T T T 1 1 2.0 T T T 1 1 3.4 T T <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> <td>4.2</td> <td>•</td> <td>622</td> <td>20</td> <td>637</td> <td>631</td> <td>101</td> <td>106</td>							4.2	•	622	20	637	631	101	106
582 68 228 897 893 0.3 B 310 0 (250) (240) 1.0 T 465 1 465 1 465 455							5.0	M	637	•	(583)	(878)	101	363
562 68 228 697 693 0.3 B 310 0 (250) (240) 1.0 T.8 B 465 1 465 1 455 456 457 457 457 457 457	_						5.3		808	(9-6)			(105)	
1.0 T 465 1 465 45	14:16	282	89	228	897		0.3	æì	310	0	(250)	(240)	144	0
859 55 557 2 610 601 2.6 T							0:1	*	465	-1	\$	455	155	8
859 55 300 896 833 0.3 B 431 0 6.6 3 851 852 851 853 651 853 653 653 653 653 653 653 653 653 653 6							r.	•	587	61	610	109	158	117
859 35 550 856 833 0.3 B 821 2 860 851 1.0 T 821 1.0 T 821 2 860 851 1.0 T 821 2 860 851 1.0 T 821 2 860 851 1.0 T 847 1 1.8 B 821 2 860 857 1 1.8 B 821 2 860 857 1 1.8 B 821 2 860 857 1 1.8 B 821 2 860 857 1 1.8 B 821 2 860 857 1 1.8 B 821 2 860 857 1 1.8 B 821 2 860 857 1 1.8 B 821 2 860 857 1 1.8 B 821 2 860 857 1 1.8 B 821 2 860 857 1 1.8 B 821 2 860 857 1 1.8 B 821 2 860 857 1 1.8 B 821 2 860 857 1 1.8 B 821 2 860 857 1 1.8 B 821 2 860 857 1 1.8 B 821 2 860 857 1 1.8 B 821 2 850 850 850 850 850 850 850 850 850 850							5.6	*	682	6 0	725	116	162	270
859 GE 300 896 833 0.3 B 850 6 (385) (375) 6 (385) (375) 6 (385) (375) 6 (385) (375) 6 (385) (375) 6 (385) (375) 6 (38		-					3.4	n	771	*	825	818	165	365
859 GE 300 896 833 0.3 B 436 (0-6) (355) (375) 1.0 T 645 (0-6) (325) 1.0 T 647 1 645 (31) 1.8 B 871 2 860 847 3 470 457 3 470 457 3 470 457 561 4.6 T 535 5 57 513 557 557 557 557 557 557 557 557 557 55							4.2	••	8	40	260	551	158	458
859 GE 300 896 833 0.3 B 431 0 (C40) (325) 1.0 T 647 1 645 631 1.8 B 871 2 860 847 3.4 B 624 4 575 561 4.6 T 635 5 57 613 5.7 T 635 6 527 (514)							5.0	ø	580	•	(385)	(375)	152	547
859 GE S00 896 833 O.3 B 431 O (C40) (325) 1.0 T 647 1 645 631 1.8 B 821 2 860 847 2.6 T 847 3 770 757 3.4 B 624 4 575 561 4.6 T 635 5 527 613 5.7 T 631 (514)							5.7	B 4	416	(0-0)			(158)	
1.0 T 645 631 1.8 B 821 2 860 847 847 3 770 757 846 4 575 861 855 85 851 851 851 851 851 851 851 851	14:27	828	30	300	896	833	0.3	2	431	0	(0+0)	(325)	213	o
B 621 2 860 847 694 694 694 694 694 694 694 694 694 694			_				1:0	*	2		279	631	252	131
## 847 3 770 757 894 4 575 861 855 851 813 853 6-6 (527) (514)					_			M	123	64	860	2.5	35	270
4 575 561 5 527 513 6 (527) (514)							2.6	84	847	60	270	157	242	411
5 527 613 6-0 (527) (514)							4.6	Ω.	769	+	575	261	232	242
(0-6) (527) (514)							4.6	4+	635	un	527	613	229	63
(9-9)				_			8.0		523	•	(527)	(214)	228	213
					_		6.1	H	637	(9-0)			(225)	

T.C. is spot welded to outside wall at indicated in, from inlet end bus location of T. C. junction on horizontal tube. B = Bottom, T = Top. Outside wall tempis by smoothing experimental date. Inside tempis by calculation, Corrected for losses. Values in () are everage over entire heated length. Bet heat to fluid up to indicated tube length.

32233

Table 87. FSSTR: DATA SUMMARY SERIES 10018-132

Heat Transfer To MCH In Miniature Heat Transfer Section

Reactor No. 10018_110; 0.0265" ID x 0.018" wall x 6" long, Type 316 S.S. Feed: MCH, 30.3 lb/hr = 7.91 x 10^6 lb/(hr·ft²)

	<u> </u>		Ex	perin	entel	Data				Ceso	thed an	d Calculated Data	
Run No.	Heagured	71.0	44	Pre		Tube '	Wall Thermoo	ouples		Vall Te	ωρ, d)	Heat ^{e)}	Cumulative ^[]
10016-	Power,	Temp,		pe			ocation	Temp,	Length, In.			Btu x 10-3	Heet, Ptu hr
-><-	her her	In ^{a)}	Out	In	Out	In. b)	Position ^{c)}	y		Outside	Inside	(hr·ft*)	hr .
14:57	778	528	538	972	1596	0.5 1.0 1.8 2.6 3.4 4.2 5.0	B T B T B	549 554 558 565 567 572 576 581	0 2 3 6 (0.6)	(547) 554 560 566 571 577 (582)	(554) 541 547 553 558 564 (569)	218 218 219 219 219 219 220 (219)	0 126 252 578 505 632 758
14:52	1,836	530	578	911	788	0.3 1.0 1.8 2.6 3.4 4.2 5.0	B T B T	600 607 620 631 638 645 653 660	0 1 2 5 6 (0.6)	(594) 609 622 633 644 653	(563) 578 591 602 613 623 (652)	516 518 520 522 523 525 525 527 ()22)	0 299 599 900 1,202 1,505 1,809
15:02	3,490	550	626	972	746	0.3 1.0 1.6 9.6 5.4 5.2 5.0	B T B T B T	671 682 707 720 762 763 762	0 1 2 3 4 5 (0.6)	(657) 685 710 730 757 762 (776)	(599) 628 65) 673 690 705 (720)	986 994 1,000 1,006 1,012 1,015 1,019 (1,005)	0 572 1,149 1,729 2,512 2,899 5,485
15:20	3,900	553	634	974	697	0.5 1.0 1.8 2.6 3.4 4.2 5.0	B T B T B T	691 704 750 763 763 771 797 806	0 1 2 3 5 6 (0_6)	(680) 707 752 756 778 797 (812)	(616) 643 668 693 715 734 (749)	1,108 1,116 1,125 1,132 1,139 1,145 1,150 (1,131)	0 643 1,290 1,942 2,600 3,260 3,920

Temp at inlet fitting. Fluid lottes on 10.6 Btu/lb before entering heated section. T.C. a most welded to outside well at indicated inches from inlet end bus bar. Loading of T.C. junction on horizontal tube. B = Bottom, T = Top. Outside well temps by smoothing experimental data. Inside temps by calculation. Corrected for losses. Values in () are average over entire heated length. Bet heat to fluid up to indicated tube length.

Table 88. FSSTR: DATA SUMMARY SERIES 10018-133

Heat Transfer To MCH In Ministure Heat Transfer Section

Reactor No. 10018-122; 0.0265" ID x 0.018" wall x 4" long, Type 316 S.S. Feed: MCH, 69.8 lb/hr = 18.23 x 10^6 lb/(hr·ft²)

	<u> </u>			aperi	nent a	1 1418				.: n	oothed an	d Calculated Date	
Run No. 10016 155-	Measured Power,		luid p, °F		ue.,		Wall Thermo	Temp.	lewth,	Ve11 7	emp, a)	Heat ^d) Flux,	Cumulative ^{e)} Heat, Rtu
•,,,,,	hr hr	In	Out	In	NIL	In. a)	thettion ^{ti})	·r'		Outside	!netde	(hr (t-) * 10-3	Rtu hr
15:51	4,710	W	204	976	49%	0.75 0.75 1.75 1.75 2.25 2.75 3.75	15 T T T T B	455 467 471 471 489 495	(0_4)	(44n) 465 475 488 (507)	(517) 552 344 559 (377)	2,020 2,030 2,030 (2,030)	0 1,165 2,554 5,510 4,690
13:6k	9,320	66	320	975	⊁94	0.25 0.75 1.25 1.75 2.25 2.75 3.25 3.75	B B T B T B	743 752 754 750 759 760 783	0 1 2 3 4 (0.4)	(758) 767 757 771 (791)	(566) 515 575 540 (561)	6,000 6,020 6,030 (6,020)	0 2,310 4,610 6,960 9,290
15:57	15,920	66	623	976	457	0.75 0.75 1.75 1.75 2.75 2.75 5.25 5.75	u B T B T	959 950 963 943 960 963 996	(Oh)	(954) 954 955 975 (1,055)	(651) 633 634 655 (718)	5,980 5,980 6,010 (6,000)	5,460 6,910 10,370 15,670
14:01	16,500	66	475	976	463	0.25 0.75 1.25 1.25 2.25 5.25 5.25	8 7 7 7 8	1,056 1,04k 1,065 1,041 1,068 1,053 1,098 1,127	0 1 2 3 1 (0.4)	(1,050) 1,050 1,051 1,082 (1,148)	(680) 680 681 713 (782)	7,150 7,150 7,150 7,150 (7,150)	4,120 . 6,240 12,360 16,530
14:30	18, 730	67	518	977	450	0.25 0.75 1.25 1.75 2.25 2.75 3.25 3.75	6 7 8 7 8	1,146 1,129 1,155 1,122 1,136 1,137 1,234 1,676	(0.4)	(1,155) 1,135 1,135 1,170 (1,600)	(729) 729 729 765 (1,215)	8,050 8,050 8,120 (8,160)	6,650 9,300 13,970 18,860
14:44	4, 770	67	510	976	496	0.25 0.75 1.25 1.75 2.25 2.75 5.25 5.75	B T B T B	465 468 477 476 484 489 502 511	(a.4)	(159) 10 10 10 195 (521)	(324) 336 346 361 (388)	2,080 2,080 2,090 (2,080)	0 1,198 2,400 5,610 4,820

The North and the state of the second

T.C.'s spot welded to outside wall a. Indicated inches from inlet ind bus bar. Location of T.C. junction on horizontal tube. B = Bottom, T = Top. Outside wall temps by emoching experimental data. Inside temps by calculation. Corrected for losses. Values in () are average over entire heated length. Net heat to fluid up to indicated tube length.

Table 89. IGNITION DELAYS FOR n-OCTANE-OXYGEN-ARCON

Press, peis	Temp,	Delay,	Press, peis	Temp,	Delay,	Press, psis	Temp,	Delay,
			80%	rgon, E	R = 0.1		·	
9.7 9.6 9.4 10.0 9.6 9.7 9.9 9.7	1218 1220 1217 1214 1130 1165 1196 1151	755 720 539 934 3500 2124 1286 2678	16.1 15.7 16.5 15.2 16.0 15.5 16.3 17.6 16.6	1208 1158 1175 1106 1164 1109 1164 1228 1167	72 777 426 3471 602 3058 672 72 496 1057	25.2 26.0 27.0 27.1 26.9	1121 1169 1163 1154 1125	3489 355 849 1270 2516
		<u> </u>	90% A	rgon, E	R = 0.1	<u> </u>		
9.3 9.2 9.3 9.5 9.6 9.6	1281 1242 1263 1242 1206 1188 1166	359 959 559 959 1462 2248 3284	15.1 15.7 15.3 15.3 15.4 15.8	1215 1265 1201 1174 1152 1147 1240	555 270 697 1585 1965 2417 369	25.2 25.1 24.7 25.6 26.0 25.5	1183 1203 1218 1169 1160 1141	1056 664 500 1611 2035 3260
			90% A	rgon, E	R = 1.0			
10.1 10.6 10.0 9.9 10.8 10.0 9.8 9.6	1221 1216 1180 1171 1252 1213 1188 1177	595 5202 3085 2930 5259 445 2946 3305	17.4 16.8 17.3 16.9 16.8 16.4 16.5	1164 1136 1159 1154 1155 1142 1160	2484 4559 4742 2039 2404 2466 2554	28.7 28.8 29.4 28.2 29.0 28.4	115% 1157 1173 1150 1179 1175	875 2551 2565 909 367 2861

(Continued)

Table 89 (Contd-1). IGNITION DELAYS FOR n-OCTANE-OXYGEN-ARGON

Press, psia	Temp,	Delay, µnec	Press, psia	Temp,	Delay, µsec	Press, peia	Temp,	Delay,
			95% Ar	gon, ER	- 0.1			
9.3 9.1 9.0 9.1 9.4 9.5	1285 1249 1268 1300 1259 1207 1195	651 968 778 457 958 1724 5181	15.8 15.1 15.6 15.9 16.1	1245 1226 1285 1187 1169 1302	773 962 358 1715 2465 327	24.8 25.5 25.5 24.0 24.8 25.1	1177 1169 1256 1175 1231 1252	1900 2276 453 886 803 517
			95% Ar	gon, ER	- 0.5			
9.8 10.2 11.3 10.1 10.5 10.0 9.9 9.8 9.8 9.7	1252 1326 1416 1288 1324 1242 1245 1258 1267 1255	2921 473 275 1473 203 3641 3644 3638 2398 2909	16.5 16.5 17.1 16.5 16.6 16.5	1275 1255 1251 1211 1253 1242 1258	334 531 2905 5120 2444 1986 399	86.9 27.1 27.1	1226 1246 1246	2307 663 1392
			95 % A r	gon, ER	= 1.0			
9.6 9.7 10.1 9.9	1230 1278 1283 1273	3404 3171 3106 3236	15.4 16.9 16.1 16.7 16.4	1220 1217 1206 1252 1250	1969 3731 3517 1712 1369	27.2 27.2 27.1	1195 1201 1205	3371 3242 1826

(Continued)

Table 89 (Contd-2). IGNITION DELAYS FOR n-OCTANE-OXYGEN-AROOM

Press, peia	Temp,	Delay,	Press, psia	Temp,	Delay,	Press,	Temp,	Delay,				
	99% Argon, ER = 0.1											
9.8 10.0 9.9 9.7 9.6 9.5 9.4	1579 1570 1421 1424 1422 1417 1458 1422	2245 2560 1001 452 1054 1226 589 957	16.0 16.0 15.9 15.9 15.6 15.1 15.0	1548 1505 1570 1554 1587 1579 1408	2294 2967 1086 1403 706 897 387	26.5 26.5 25.8 25.2 24.9 25.0	1550 1502 1536 1541 1592 1595	2222 2841 1144 954 418 386				
	L		99\$ Az	gon, KR	- 1.0		····					
9.8 9.9 2.8 9.5 9.5	1335 1362 1394 1397 1403 1348	3283 1295 651 521 457 1743	15.2 15.9 16.2 16.0 15.9 16.4	1282 1357 1362 1398 1390 1359	3501 1164 874 326 456 906 1612	26.7 26.1 24.9 25.6 27.8	1323 1334 1348 1391 1305	1188 965 516 65 2559				
			99\$ Ar	gon, ER	- 1.9							
10.0 9.9 10.1 10.5 10.1 10.5 10.2	1338 1363 1459 1436 1440 1423 1331 1301	2575 2291 400 1195 664 1291 2602 5037	16.5 16.5 16.4 15.9 15.9 16.7 16.5	1346 1310 1413 1413 1425 1397 1397	2479 2655 661 562 530 1186 2164	27.1 27.6 26.8 25.8 25.4 25.1 25.0	1330 1290 1383 1391 1440 1389 1393 1405	2471 2107 1150 124 166 190 190				

Table 90. IGNITION DELAYS FOR DECALIN-OXYGEN-ARGON

Press, psis	Temp,	Delay,	Press,	Temp,	Delay,	Press, peia	Temp,	Delay,
			80% Ar	goa, ER	- 0.1	·		<u> </u>
10.4 10.5 10.5 10.5	1218 1251 1261 1295 1201	1743 1040 561 324 3627	17.4 17.5 18.5 18.5 18.6	1243 1217 1206 1178 1162	439 753 1106 2349 3895	28.9 29.4 29.4 29.9 29.9 30.0 29.9	1255 1193 1204 1157 1185 1172 1156	159 708 474 2956 980 1211 3887
	\ 	<u></u>	90% Ar	gon, ER	- 0.1	<u> </u>		L
9.3 9.1 9.2 9.1 9.5 9.6	1224 1251 1266 1241 1259 1196 1182	1402 1204 405 905 938 1987 3299	14.6 14.7 14.7 15.1 15.6 15.7	1185 1220 1247 1180 1177 1158	1321 667 369 1781 1977 2686	24.6 24.2 24.1 25.0 25.6 25.8	1187 1200 1226 1165 1164 1154	958 663 334 1706 1444 2225
		····	90\$ Ar	gon, ER	- 0.2			
10.7 9.7 9.6 9.8 9.8 9.7	1247 1244 1269 1301 1135 1189 1025	1157 1088 582 276 4015 4019 3499	16.0 15.9 15.8 16.4 16.5 16.4 16.5	1257 1197 1225 1168 1201 1165 1205 1178	137 672 541 3329 672 4346 639 2738	26.4 26.5 27.1	1198 1176 1172	369 601 2199
			95\$ Are	on, ER	= 0.1			
8.8 8.9 8.9 8.9 9.1 8.7 9.0	1200 1245 1250 1275 1325 1270 1308	5186 1611 1926 779 230 845 262	15.1 15.2 15.2 15.4 15.6 15.7 15.9 16.1	1255 1250 1218 1248 1246 1255 1205 1187	452 548 1569 516 645 932 1913 2222	25.6 26.0 25.6 25.9 31.6 36.0	1235 1220 1219 1190 1190 1170	514 1409 961 2224 2542 3161

(Continued)

Table 90 (Contd-1). IGNITION DELAYS FOR DECALIN-OXYGEN-ARGON

Press, peia	Tomp,	Delay,	Press, pein	Temp,	Delmy, µmec	Press, peia	Temp,	Delay,			
	95\$ Argon, FR = 0.2										
9.4 9.0 9.2 9.9 9.7 9.1	1260 125k 1300 1250 1222 1252	1075 1203 362 1820 3678 1560	15.4 15.8 16.2 15.8 16.5	1267 1253 1258 1228 1177 1223	457 845 1231 1971 3826 1874	25.9 25.6 26.5 26.5 26.9 26.9	1244 1266 1246 1226 1220 1219 1211	1006 326 649 1809 1935 1806 2057			
			99\$ Ar	gon, ER	= 0.2						
8.8 8.7 8.7 9.0 9.1	1265 1282 1317 1385 1388	2195 1448 1078 354 322	15.2 15.3 15.2 15.4 16.2 15.9	1321 1302 1265 1249 1247 1214	444 632 1254 1593 2092 2850	25.6 25.0 24.8 25.6 26.0 26.2	1266 1271 1295 1234 1224 1207	847 785 442 1681 2112 2845			
			99% Ar	gon, ER	= 0.5						
9.8 9.4 9.5 9.1 9.0 9.9	1574 1556 1580 1595 1415 1320 1349	1063 1444 903 711 325 3633 2179	15.7 15.5 15.5 15.1 16.2 16.5	1322 1337 1372 1360 1311 1296	1466 1247 515 707 2227 2726	26.6 26.0 25.2 25.4 25.5 26.5 26.8	1327 1336 1348 1376 1361 1281 1276	1404 1215 609 193 321 2212 2683			
			99% Ar	gon, ER	= 1.0						
9.9 10.0 9.8 9.6 9.5 9.6	1341 1384 1393 1420 1431 1453	3223 2860 2149 1571 1115 395	16.5 15.9 15.8 15.7 16.6 16.4	1398 1395 1420 1407 1356 1363	1467 977 458 653 2586 2137	27.4 27.3 26.9 26.3 25.8 26.0	1325 1347 1364 1388 1397 1422	2954 2130 1749 1106 717 393			

Table 91. ICHITION DELAYS FOR "SHELLDYNE"-OXYGEN-AROON

* Delay times for early ignition.

[] Delay times for two ignition points on the same shock tube run.

Press, psia	Temp,	Delay,	Fress,	Temp,	Delay,	Press, paia	Temp,	Delay,				
	90% Argen. ER = 0.1											
			15.9 14.5 14.5 15.7 14.1 14.2 14.8 15.8 15.0 15.9	1426 1400 1341 1280 1212 1178 1215 1193 1180 1361 1484 1516	155 214 528 643 965** 2255** 1064** 2032** 249 82 554							
			95 % A	rgon, E	R = 0.2							
8.5 8.4 8.5 8.1 8.5 8.8 8.8 8.9 8.8 8.9 8.8 8.9 8.8	1456 1456 1471 1432 1449 1563 1463 1443 1412 1453 1375 1312 1291 1252 1291 1332	531 1939 542 1307 546 J 874 1444 610 571 567 5434 7854 2450 9839 2189 1703	14.0 14.7 14.5 14.0 14.7 14.8 15.2 15.5 15.7 16.1 16.0	1378 1466 1480 1478 1574 1569 1377 1372 1328 1279 1322 1312 1769	833 216 224 187 104 110 502 884 1040 2546 1387 1609 3006	25.5 24.1 24.1 25.6 24.8 25.5 25.5 25.5 25.5	1344 1414 1387 1372 1325 1331 1327 1287 1267 1253 1249 1249	533 191 226 360 832 818 880 938 1514 3134 2896 2559				

Table 91 (Contd-1). IGNITION DELAYS FOR "SHELLDYNE"-OXYGEN-AROON

Press, pein	Temp,	Delay,	Press, psia	Temp,	Nelay,	Press, psia	Temp,	Delay,
			991m A	rgon, I	R = 0.1			
9.778.8 8.8 9.9	1433 1498 1472 1447 1478 1461	2096 611* 843* 1808 981* 1946	14.9 15.2 14.9 15.2 15.3 15.4 15.7 15.5 14.5 15.7 15.7 15.7 15.7 15.7 15.7 15.7	1446 1456 1456 1451 1518 1558 1558 1459 1451 1456 1554 1456 1554 1456 1456 1456	850* 1578 1578 1200	24.7 24.4 25.1 25.0 24.6 26.9 24.5 25.6 26.8	1475 1495 1454 1490 1371 1405 1427 1350 1370	649 406 726 1461 1433 2629 2280 3336 4217

Tatle 92. IGNITION DELAYS FOR "SHELLDYNE" HOXI GEN-ARCON-MIXTURES

Press, peia	Темр,	Delay,	Press, peia	Temp,	Delmy,	Press, psia	Temp,	Colay,
			80% A	rgon, E	R = 0.1	4		
	, ,		14.2 14.6 15.3 15.1	1261 1247 1245 1191	331 339 256 1507	24.0 24.1 23.7 24.3 24.2 24.7	1264 1209 1201 1201 1151 1147	321 92 601 785 1539 3195
			95 % A	rgon, E	R = 0.1			
			14.7 14.6 14.0	1470 1428 1355	108 136 308	24.6 24.5 24.5 24.1 24.1 25.9	1537 1472 1450 1447 1382 1312 1265 1217 1257	59 65 156 84 99 365 730 1715 676
			95 % A r	gon, ER	= 0.2			
9.5 8.5 9.1 8.6 9.0 9.2 9.7	1425 1511 1592 1525 1511 1291 1286	228 329 368 572 953 1196 3622	14.3 14.9 14.9 15.2 14.8 14.7 15.1 15.2 15.6	1432 1514 1412 1376 1408 1354 1334 1238 1246 1225	150 75 145 241 155 269 362 676 1485 3403	24.5 24.7 25.3 24.4	1386 1366 1360 1271	206 183 231 777

(Continued)

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Table 92 (Conta-1). IGNITION DELAYS FOR "SHELLDYNE" H-OXXGUH-ARCOH-MIXTURES

Press, pula	Temp,	Delay,	Press, psia	Temp,	Delay,	Press, psia	Temp,	Delay,			
	98% Argon, ER = 0.2										
6.6 8.7 8.8 9.0 9.0	1285 1717 1677 1667 1611 1590	1945 123 132 145 209 215	14.9 15.1 15.0 15.0 14.8 15.4	1519 1567 1456 1393 1295 1385	247 155 368 551 3033 557	23.7 24.2 22.9 24.1 24.1 24.6	1466 1450 1349 1418 1341 1303 1256	175 222 512 261 489 1092 2445			
	98% Argon, ER = 0.5										
8.6 9.0 9.5 9.0 9.4 8.7 9.3 9.3	1470 1583 1593 1466 1418 1465 1382 1425 1376 1372 1372	382 167 180 612 639 478 804 432 947 1154 2867	14.4 14.9 15.4 15.0 15.2 15.0 15.7	1505 1525 1507 1433 1397 1295 1295	220 191 200 328 471 1602 1716	24.1 24.4 24.0 24.1 24.9	1461 1442 1377 1331 1280 1261	183 236 463 747 1580 3046			

Table 93. IGNITION DELAYS FOR DATE-OXYCEN-ARCON

Press, psia	Temp,	Delay,	Press,	Temp,	Delay,	Press, psia	Terip,	haec Cejul,
			80% A	gon, ER	= 0.1			
11.0 10.1 10.1 10.0 10.4 10.2 9.6 9.6	1553 1436 1432 1436 1394 1331 1225 1258	282 388 395 484 486 910 3969 3122	15.3 15.5 14.7 15.2 15.9 16.0 15.9	1351 1393 1283 1292 1287 1248 1187 1250	219 80 278 332 884 466 3768 912	25.6 23.9 24.7 26.0 26.0 26.3	1516 1228 1250 1225 1161 1208	92 249 495 660 3699 1329
			90% Ar	gon, ER	= 0.1	<u> </u>		
8.8 8.9 9.2 9.5 9.7 9.7	1408 1422 1421 1398 1338 1331	252 179 253 419 1096 4051	14.5 15.6 15.2 14.8 15.4 16.2 16.4	1346 1453 1418 1307 1285 1293 1256 1191	268 112 98 937 1022 959 1268 3855	25.8 24.5 25.2 25.6 26.2 25.7 26.6	1403 1520 1521 1507 1256 1195 1247	117 285 917 432 1079 3634 2061
			90% Ar	goa, ER	= 0.2			
9.4 9.2 9.6 9.9 10.4	1419 1425 1388 1342 1334	226 175 468 1003 891	15.2 15.8 15.8 15.9 15.8 16.8 16.7	1384 1449 1378 1333 1282 1269 1215	261 127 230 331 1036 939 3348	25.3 26.6 26.3 25.3 26.5 27.3	1369 1388 1349 1260 1255 1251	186 180 266 777 937 1962

P. P. SECONDARY SECONDARY OF SE

Table 94. IGNITION DELAYS FOR "SHRILDYNE"-DECALIN-OXYGEN-ARGON

Press, peis	Temp,	Delay, µmeo	Press, psia	Temp,	Delay,	Press, peia	Temp,	Delay, µsec
			90% Ar	gon, ER	- 0.1			
9.4 9.3 9.7 9.7	1350 1 263 1254 1263	357 1037 1916 1057	14.5 15.8 14.7 14.3 15.8 15.8 16.1 15.2	105 1196 1162 1201 1198 1223	210 486 649 1257 1817 3303 3269 1429	31.5 24.8 25.0 24.8	1338 1290 1288 1195	122 316 674 1244
			95 % A r	gon, ER	= 0.2			
9.6 8.9 9.2 9.1 9.5 10.1	1164 1361 1358 1301 1297 1319	180 583 599 1298 1800 3219	15.0 14.6 15.3 14.7 15.4 15.6 15.4	1425 1366 1384 1304 1320 1302 1241 1214	146 500 354 951 571 770 1419 3351	25.1 24.4 24.0 24.7 25.9 25.6	1415 1354 1305 1283 1281 1247	107 176 486 635 1429 3247
			98% Ar	gon, ER	- 0.2			
9.1 8.7 9.1	1516 1370 1422	289 1116 479	14.4 14.6 14.7 14.8 15.5	1407 1455 1385 1338 1320 1277	305 244 241 727 1192 1594	25.0 24.5 24.5 25.0	1391 1357 1322 1272 1278	200 314 706 2870 1186
			99% Ar	gon, FR	~ 1.0			
9.5 9.5 9.2 8.8 9.0	1493 1563 1598 1601 1679	3264 2686 730 486 327	15.2 15.3 15.2 15.2 14.9 14.9 15.6 18.8	1471 1550 1497 1535 1553 1590 1690 1724	2550 776 2732 964 925 459 218 96	24.4 24.7 25.0 24.6 24.5	1416 1500 1585 1545 1523	2687 804 250 446 708

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Table 95. IGNITION DELAYS FOR "SHELLDYNE"-BINGR S-OXYGEN-ARGON

Press, psia	Temp,	Delay,	Press, psia	Temp,	Delay,	Press, psia	Temp,	Delcy,
			95% Ar	gon, ER	= 0.2	·		
8.6 8.3 6.3	1346 1436 1273	386 323 3174	13.9 14.1 14.5 14.6 14.8 15.1	1339 1382 1340 1267 1220 1268	274 213 421 1271 2667 1556	23.5 24.1 23.2 22.9 23.9 23.8 24.3	1346 1343 1267 1212 1276 1265 1268	275 287 770 2755 847 1025 1017
		_	99% Ar	gon, ER	1.0			
9.1	1777	170	14.3 15.0 15.2 15.3 15.1 15.9	1466 1597 1572 1476 1399 1378	85 ¹ 4 186 393 651 1165 25 ¹ 41	24.5 24.5 24.3 24.1 25.1 25.9 25.8	1557 1523 1479 1418 1404 1356 1384	178 283 415 716 1182 2569 1934

Table 96. CHARACTERISTIC PROPERTIES OF F-71

Hydrogen-Carbon Ratio	2.16
Molecular Weight	177.5
Critical Temperature	737 °F
Critical Pressure	252 psia
Critical Volume	0.0683 ft ³ /1b
Helting Point	-51 °F
Heat of Combustion	18930 Btu/1b
Normal Boiling Range	420-543 °F

Table 97. LICUID PROPERTIES OF F-71 AT SATURATION PRESSURE

Teoperature, or	Heat of Vaporization, Blu/15	Yapor Pressure, pela	Specific Heat, Btu/lb-F	Density, Ib/ft ³	Viscosity, 16/ft-hr	Thermal Conductivity, Otu/ft-hr*F	Enthelpy, Stu/Ib
0	157.7	0.0	0.344	50.7	6.85	0.0417	-181.7
100	148.7	0.005	0.412	48.4	2.50	0.0455	-140.7
200	138.0	0.147	0.471	45.9	1.23	0.0483	- 93.0
300	127.7	i.59	0.522	43.2	0.734	0,0493	- 38.6
400	111.6	8.76	0.569	40.1	0.494	0.0487	21.3
500	8.69	31.3	0.615	36.7	0.354	0.0468	85.8
600	78.1	84.4	0.674	32.3	0.259	0.0434	155.8
700	44.4	192.0	0.876	25.5	0.175	0.04	733.6

Table 98. GAS PROPERTIES OF F-71

-										- 0		-,					
	,	1.00	m	9 10	6.97	We.	610	Fra	Am		1700	1100	מיתי	130	1 LAK	15.00	1A-20
1 Au 19 u 19 u 19 u 19 u 19 u 19 u 19 u 19	C 5.73 () US () () US	0 1414 0 119 0 19 0 19 1 19 1 19 1 19 1 19 1 1	n nag d hyp d the d that d that d that d that	0 % 4 0 mm 0 1 % 8 0 (6 1) 0 .6-7 0 .8-46	الاحداد ال	Ulina? E ne/ P ne O see O see	7,07% 6,015 7,317 6,437 0,438	1 20	0.28 M 0.06 M 0.06 M	1,34A	0 1145 0 1741 0 0721 0 0721	0.1277 6 /540 0 2777 0 dlass	6.161k 3.70# 0.771;	6 11. ⁵⁴ 6 u/da 6. 56?	8,1212 0,094 0,064	9.15u) 0.1.nd 0.1886	0.177 0.11.6 0.172
1300 1300 1100 1100 1000 1000 1000 1000	0 (1%) 0 (1%) 0 (1%) 0 (1%) 0 (1%) 0 (1%) 0 (1%) 0 (1%)	3.0% 6 0% 9 0% 3 144 0.0%	0 9500 0.0 mf 2.0377 0 mile 0 mile 0 mile	0,0616 0,04m 0,04m 0,040 0,040 0,040 0,0516	C 477	0.719	3 32 40	0.1964 0.121 0.121 0.121 0.121 0.121	0 08/8 0.770 0 (77) 0 77-2	G mijA G mer i CN i	O JOHN Copyri Copyri Copyr	0.045	9.1700 9.1%-a (0801	3.1.7% 0 77, J.061	0.1 1 0.1640 C.JPAZ	0.1136 7.1036 0.1036	3-3168 9-1117 9-2078
1900 1900 1900 1900 1900 1900 1900 1900	0 966 0 676 0 1 1 0 677 0 663 0 635 0 663 0 579 0 771	0 649 0 679 0 71 0 71 0 719 0 719	9 6 at 0 ftps 0 ft7 0.75 0.75 0.75 0.75	0.86y 0.745 0.747 0.746 0.746 0.746	0.76A 0.777 0.796 0.758 0.772 0.79	0.862 0.921 2.765 0.772 0.779	0. app 0. Pol 0. Pp 0. Pp 0. Pp 0. Pol	0.F31 0.e36 0.747 0.744 0.609	0 Fg 0 Jg 0 Jg	0 945 6,600 6,600 6,606 0 8,5	0.618 v.300 0.610 0.614	0. 100 0.307 0.636 0.625	0.804 0.01 0.03	6.814 6.825 c,832	n,fas o.rui o.eys	0.797 0.24 0.65	0. 191 0. 021 0. 833
100	.1.1			Γ				ı —	Eatha 1	#. B1w/15						1	
700 941 643 643 749 644 1406 1406 1406 1406	85.8 89.5 157.5 157.5 266.6 519.5 85.5 85.7 857.4 867.4 868.2 718.7	344.3 626.6 641.3 744.6 647.4 313.8	369.8 615.8 646.3 609.6 TI2.6	J.m. 4 o.e. r o.e. r o.e. 1 o.e. 308.5 697.0 697.6 612.5 428.8 707.1	544.5 671.9 941.5 665.6 906.3	162.3 brd.3 546.3 628.8 701.7	1 141.5 240.6 40.6 40.7	\$19.4 316.7 610.3 6#.8	5 m.9	LM.6 77.8 611.0 6N.6	635.6 546.5 608.5 590.8	505.8 606.1 606.4	521.7 603.9 686.8	5.80.1 601.9 601.6	518.9 600.5 645.2	518.0 518.8 681.7	
910 900 1000 1100 1200 130	0.95 6.95 1.050 1.08 1.146 1.14	0,730 0,967 1,059 1,059 1,137 1,146	0. #1 0.977 1.630 1.491 1.130 1.159	0.00 0.00 1.00 1.00 1.00 1.12 1.12	0.861 0.977 1.016 1.869 1.111 1.148	0.71 1.010 1.025 1.319 1.357	0.442 1.005 1.360 1.119 1.361	0.936 1.000 1.036 1.108 1.158	0.996 1.093 1.138 1-136	8.998 8.050 1.103 1.1;3	0.766 1.067 1.100 1.151	0.487 1.34 1.05 1.169	1.0% 1.0% 1.119	113	1.098 1.098 1.146	1.037 1.030 1.344	1.055 1.050 1.161
7340 1700 1700 1700 900 900	0. 0. 0. 0.	1,699 6-117 1,218 1,136 1,099 J-770	3.46 3.30 4.45 4.37 4.36 8.00	1422 122 117 118	93.81 8.39 6.29 5.25 6.65 6.34	11.41 6.61 6.00 3.50	17-15 10.80 8 az 7-38 6-34	22.30 13.57 10.37 8.78 7.72		17.19 16.26 11.17 10.66	21.56 15.91 11.59 11.50	22.79 17.75 16.30 17.56	19.55 11.7 15.46	20.45 17.00 16.57	21.47 M.20 15.65	67,69 19,20 18,65	22 M 20 20 21-57
1323 2371 1326 1326 687 687	1. 4. 1. 1. 1.	0.475 0.909 0.447 0.946 0.946	0 717 0 806 0 809 2 800 0 812 0 80	0.516 0.701 0.701 0.700 0.456 0.456	0.741 6.546 0.777 0.803 0.815 2.815	0.416 0.61 0.61 0.61 0.61 0.61	0.625 0.737 0.610 0.863	0. 101 0. 101 0. 11- 0. 11- 0. 101	0.557 0.67 0.762 0.84	6.191 6.679 6.773 7.600	0,590 0.666 3,966 0.657	0 1 1 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2	8.451 8.756 9.636	8.647 0.760 6.637	0.64m 0.74h 0.641	0.706 0.776 0.667	0.750 0.750 0.6;
840 955 1996 1170 1270 1380	\$.018 \$.219 \$.016 \$.75 \$.019	1.0k) 3.038 1.096 1.029 i VO 1.019	1.146 1.062 1.062 1.033 1.077 1.077	1,544 1,1,57 1,007 1,366 1,036 1,030	i per i per i ins i ins i ins i ins i ins i ins	1.444 5.154 1.056 1.057 1.065	3,173 1,298 1 105 1,067 1,069	1 %9 1-127 1-076 1-049	1.659 1.152 1.058 1.058	1.167 1-161 1 160 1.06A	1,6%; 1,4%; 1,1%; 1,0%;						
800 900 1001 1301 1301 1302	600 631 6-1 6-1 6-1 7-16	80-1	4 377 1009 1461 6403 6 35 6 10	376 646 371 371 470 696	118 146	817 879 787 630 645	599	67) 137 138 683	100 100 179 619	552 546 608 651	917 959 610 640						
8x0 900 1000 1:x0 1300 1300	0.1245 0.0754 0.0815 0.0815 0.0877 6.0579	0.12% 0.07 P 0 PB TO 0.75 W 0.05 PB 0.05 PB	0.1618 0.1177 0.0777 0.041 0.041 0.0376	0.2 9V 0.11.6 0.0717 0.0719 0.0440 0.0367	0.1171 6 (75) 0.7840 0.4394	0.1176 0.0736 0.0743 0.11439 0.0338	0, 10#4 0.0719 0.090N 0.039A 0.0319	0.06% 0.06% 0.03% 0.03%	0.0(2) 0 (6.5) 0 (2)(4) 0 (2)(4)	0,0° y8 8 .%25 8 0550 0.0066	0,094) 0,1647 0,0114 0,015						

AFAPL-TR-67-114 Part II

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Table 99. CHARACTERISTIC PROPERTIES OF TRANS-DECALIN

Molecular Weight 138.3 Critical Temperature 767°F Critical Pressure 422 psia Critical Volume 0.0565 ft³/1b Heat of Combustion 18,289 Btu/lb

Table 100. LIQUID PROPERTIES OF TRANS-DECALIN AT SATURATION PRESSURE

Temperature, T	Heat of Yaporization, Btu/lb	Yapor Pressure, pela	Specific Heat, Btu/lb-"F	Denotity, 1b/ft ³	Viscosity, 1b/ft-hr	Thermal Conductivity, Btw/ft-hr°F	Enthalpy, Btu/Ib
0	150.3	0.001	0.366	56.3	3.74	0.054	-173.3
100	143.2	0.065	0.423	53.8	1.679	0.058	-135.5
200	135.2	0.905	0.475	51.1	0.967	0.061	- 90.8
300	126.0	5.75	0.520	48.2	0.642	0.062	- 40.4
400	115.1	22.3	0.563	45.1	0.464	0.061	14.3
500	101.8	62.5	0.606	41.6	0.352	0.059	72.6
003	84.6	141.5	0.660	37.4	0.271	0.058	135.8
700	58.9	278.0	0.784	31.6	0.201	0.053	206,7

Table 101. GAS PROPERTIES OF TRANS-DECALIN

					p.	900 UFO . p-	10				
\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \	,	100	700	100	600	570	6·10	7a	ho	Yr.	1
_					Attent	g . 31 ₄ 75-4	Ÿ.		*****************		
9440 900 900 900 1000 1130	0.045 0.045 0.045 0.052 0.054 0.057 0.057 0.065	0.05/7 0.057 0.076 0.077 0.06/3	0.0%3 5.6375 0.6574 0.10-75 0.0000	0.0346 9,0616 9,068 0,0636	0,0696 0,0695 0,051 0,0673	C, IFV6 0,046 0,7473 C, IRa6	0.18]8 0.0919 6.08 8 0.7508	0.05.17 3.75.18 0.05.16	g. QL12 g. 2 An g. U555	0,1764 3,3746 0,1766	0,144.7 0,173.96 0,06.98
[Theren	1 Conducti	etti, kud	ft-hr-"f				
400 500 600 706 800 900 1000 1100	0.016 0.0177 0.208 0.045 0.045 0.057 0.057	0,012† 0,025† 0,034 0,037 0,036 0,036	0.04.07 0.036 0.2369 0.1746 0.0423	3.0%1 0.0577 0.0610 0.063	G.wog G.thie G.Shig G.Oh?z	0.7641 0.06 5 0.0475 0.0522	0.10% 0.75% 0.056 0.056	0.0634 0.007 0.007 0.007	7, 1989 0.0636 0.9615	0,1015 9,06 +5 1,085 9	0.100 0.0173 0.0173
١.	1	γ	т		ibectitie in	AL BLU/10				 -	γ
1100 1100 1000 1000 1000 1000 1000 100	0.276 0.339 0.345 0.676 0.518 0.511 0.569 0.596 0.659 0.619	0.575 0.569 0.659 0.655 0.675	0.6-9 0.6-8 0.643 0.662 0.662	0.664 0.654 0.671 0.640	0.768 0.603 0.643 0.677	1.90; 0.724 0.643 0.706 7, Ptu/18	0.667 3.799 0.718 6.719	C 616 0.740 0.746	3.004 0.765 0.777	0.877 0.730 0.736	0.7% 0.618 0.7°7
١.		1	τ	Υ	ERVIAL;	7, 1410		1			Y
11.00	~25.0 7.8 86.5 152.5 161.6 296.5 296.5 968.3 407.2 472.8 536.9	225.0 175.1 182.9 189.9 169.2 535.6	274.1 336.7 600.1 663.3 532.6	520.9 54.6 61.2 5-7.2	317.9 90.6 636.8 525.7	##C.1 9f1.6 552.0 522.1	276.3 377.9 646.9 518.6	363.2 141.6 314.6	310.0 530.0 510.0	363.0 696.0 507.1	798.6 798.6 175.8 175.8
			,	,	Entrapy.	Blwib-'P	· 	···			
900 900 1000 1100	0.760 0.830 0.876 0.924 0.918 1.015	0.755 0.747 0.854 0.959 0.959	0.761 0.653 0.661 0.928	0.862 0.673 0.965	0.811 0.863 0.913 0.979	0.759 0.857 0.907 0.908	0.77% 0.649 0.90£ 0.94y	0.841 0.897 0.441	0.839 0.648 0.941	0.844 0.867 0.956	0.921 0.803 0.936
600		1.458		,	Pennit	y, 19/11	,	ı			
1700 1000 800 800	0.00.00	1.259 1.116 1.009 0.966 0.651	2.18 2.16 2.17 1.950 1.771	4,27 3,55 3,09 2,70	7.48 5+ 6.36 3.86	15.79 7.51 5.54 5.05	27.74 10.08 1.59 2.59	19.79 9.39 7.64	16.79 11.62 9 or	29.16 15.67 10.56	27.55 16.11 12.13
		0.89			Compressio	1114 Per	o r i				,
900 900 900 1000 1100	1. 1. 1. 1.	0.8% c.867 0.918 0.959 0.953	0.765 0.676 0.676 0.905 0.927	0.718 0.604 0.84 0.871	0.578 0.779 0.906 0.855	0. 934 0.648 0.796 0.853	0.222 0.544 0.707 0.706	0,643 0,861 0,777	0.405 0.418 0.729	0.501 0.501 0.704	6 947 0.947 0.661
l}					atho of Sp	orifie Nova	10				,
17.6 1000 600 600 600	1.027 1.096 1.025 1.026 1.028	1.082 1.052 1.061 1.0% 1.090 1.028	1.197 1.075 1.051 1.061 1.095	1.16. 1.07: 1.0 † 1.165	1.646 1.139 1.080 1.057	1.253	1.521 1.160 1.091	9.9k5 1.221 1.115	1.90k 1-139	1.418	1.948 1.201
600 700 800 900 1000 1100	626 634 661 706 733 737	177 384 629 641 701 727	570 624 663 706	. Se	18 18 131 145 640	891 377 640	623 153 679	505 536 610	528 601	546 195	536 578
600	0 12 00	0.,070	<u> </u>	1919-	Thumbon Cr	FIFICIONS,	17/301		Γ	ı ——	
900 1/100 1/100 1/100	0.1577 0.1107 0.8% 0.0% 0.0539 0.0539	0.3070 0.1316 0.0984 0.0713 0.0749 0.0846	0.1%3 0.10m/ 0.0779 0.0601 0.0681	0.1970 0.0073 0.0699	0.1764 0.0674 0.0574 0.0513	0.1001 0.0712 0.0520	0.3155 0.0727 0.0525	0.2180 0.0724 0.0519	0.0510 0.0101	6,0/85 0,000	0.66°8 0.0%7

AFAPL-TR-67-114 Part II

Table 102. CHARACTERISTIC PROPERTIES OF "SHELLDYNE" LI

Critical Temperature 945.4 °F Critical Pressure 448 paia Critical Volume 0.0478 ft 1/1b Heat of Combustion 17845 Btu/lb Normal Boiling Point 501.8 °F

Table 103. LIQUID PROPERTIES OF "SHELLDYNE" AT SATURATION PRESSURE

Temp,	Density, lb/ou ft	Viscosity, lb/ft/hr	Thermal Conductivity, Btw/hr/ft/F	Surface Tension pndla/ft	Heat Capacity, Btu/lb/°F	Heat Content, Btu/1b	Heat of Vaporization, Btu/lb	Vapor Pressure, paia
-60	72.07	≟20 00.	.098	,0994	.207	-13.6	144.7	-
-40	71.53	4850.	.097	.0970	1550	-9.3	143.6	٠ -
-20	70.99	1405.	.097	.0,446	.232	-4,8_	142.5	
0	70.45	552.	.0· y6	.0922	.245	0.0	141.4	} ~
20	69.91	167.	.095	.0898	,257	5.0	140.5	,0000
40	69.37	108.	.095	.0874	.270	10.3	159.1	.0001
60 30	68.82	72.9	•03/4	.0851	.282	15.8	157.9	.0002
<u> </u>	68.27	50.4 35.7	.094	.0827	. 307	21.6	136.7 135.5	.0006
120	67.72 67.16	25.9	.692	.0781		33.9	134.3	.0033
140	66.60	19.2	.092	.0758	.319 .352	40.4	135.1	.0071
160	66.04	14.5	.091	.0735	. 544	47.1	131.8	.0144
180	65.47	11.2	.090	.0712	356	54.1	130.5	.0277
200	64.89	8.78	.090	.0689	.356	61.4	129.2	.0510
220	64.31	6.96	ا وران	.0667	.381	68.9	127.9	.0900
240	63.72	5.60	.088	.0644	.393 .406	76.6	126.5	.1528
260	63.13	4.56	.087	.0622	.406	84.6	125.1	.2505
280	62.53	3.75	.086	.0600	.418	92.8	123.7	.3976
500	61.92	3.12	.085	.0578	.43C	101.5	122.3	.6129
520	61.51	2.61	.084	.0556	.441	110.0	120.9	9199
540	60.69	2.21	.083	.0535	.452	119.0	119.4	1.347
360	60.07	1.89	.082	.0513	.463	128.1	117.9	1.928
380 400	59.43	1,62	.080	.0471	.473	137.5	116.3	2.703
400	58.79	1.41	·(7·;	.0471	.495	147.0	114.7	3.718
440	58.13 57.47	1.23	.078 .076	.0429	.495	156.8 166.8	113.i	5.023
460	56.80	1.07 .952	.075	.0408	.503 .513	176.9	111.5 109.8	6.677 8.743
480	56.11	.844	.075	.0388	522	187.3	108.0	11.28
500	55.42	752	.072	.0367	.532		106.2	14.38
520	54.72	.752 .674	.070 l	.0347	.541	197.8 208.6	104.4	18.11
540	53.99	.619	.069	.0327	.550	219.5	102.5	22.56
560	53.25	.573	.067	.0308	.559	250.5	100.6	27.81
580	52.50	.55 <u>1</u>	.066	.0288	.568	241.8	98.5	33.95 41.09
600	51.73	.492	.064	.0269	.577 .586	253.5	96.5	41.09
620	50.94	457	.065	.0250	.586	264.9	94.5	49.32
640	50.13	424	.061	.0232	.594	276.7	92.0	58.75
660	49.29	. 394	.060	.0213	.603	288.7	89.7	69.48
680	48.43	. <u>%7</u> .741	.058		.611	300.8	87.5	81.63
700	46.61	.518	.057 .055	.0177	.620 .629	313.1 325.6	84.7 32.0	35.51 110.6
740	45.65	.296	.053	.0143	.638	338.2	79.2	110.0
760	44.63	.276	.052	.0126	.648	351.1	76.1	127.7 146.8
780	43.57	256	.050	.0109	658	364.2	72,9	167.9
800	42.43		,050 ,043	.0034	.669	377.4	69.4	191.5
820	41.22	.221	.046	.0078	.682	500.9	65.6	217.1
8110	59.89	.205	.045	.0063	.597	300.9 404.7	61.4	245.5
oéo	38.43	.189	.043	.0049	.725	418.8	56.7	276.7
880	36.76	.172	041	.0035	.740	437.4	51.3 44.6	310.9
900	34.77	.155	.040	.0025	.779	448.5	44.6	346.5
920	32.17	-135	,039	.0011	.863	464.8	35.8	389.8
940	27.50	.104	.056	.0002	1.537	484.7	19.9	435.2

a) 32.2 poundals = 1 1b.

Table 104. CAS PROPERTIES OF "SHELLDYNE"

Year,	Pressure, pale													
'r'	0	100	200	140	600	800	1000	1200	1400	1600	1.100			
					Path	alpo, al Bru/l	b							
-100 100 300 400 500 600 700 600 1000 1100	124,6 181,1 165,1 190,6 273,8 963,4 905,9 953,9 860,6 87,0,6 671,8 710,8 770,8	454.6 514.2 576.4 640.8 707.4	906.0 971.5 656.9 704.1 775.1	556.1 627.4 597.6 757.1	\$26.0 613.7 681.6 740.5	326.7 396.1 e17.6 13.12	513.3 590.8 609.8 746.4	511.6 567.2 664.1 741.2	510.2 585.1 661.0 737.5	509.4 503.7 699.0 735.0	50 ⁶ .9 502.8 697.6 733.2			
1500 1500 1500 1500 1700 1700	847.7 918.5 950.4 1000.9	775.9 665.5 916.5 986.6 1061.5	043.1 914.5 906.7 4059.6 1199.9	767.1 838.1 910.1 983.0 1056.2 1129.9	832 7 905.7 979.1 1052.9 1126.9	621.2 901.2 976.2 1049.8 1323.9	891.6 896.8 971.6 1066,6 1121.1	617.2 892.9 968.2 1045.4 1338.1	813.5 889.5 963.8 1040.7 1116.1	810.8 :86.8 962.7 1098.4 1114.0	808.8 884.8 950.7 1035.5 1112.2			
		Pagecit; pata												
900 1000 1100 1200 1300 1400 1500 1600 1700			166.4 173.3 179.2 185.8 187.6 190.5 192.7 194.0 197.1	294.9 317.5 335.6 350.3 362.0 371.1 370.3 364.0 360.7	759.1 416.0 456.6 489.1 513.1 733.6 751.8 764.7 777.1	396.5 460.1 550.0 605.0 651.3 667.3 713.9 736.8 757.8	483.0 520.9 684.0 703.3 773.0 887.9 871.9 907.8 935.7	455.1 373.3 688.6 792.9 883.6 959.3 1021.3 1071.8 1111.6	481.3 616.2 749.1 874.2 966.8 1004.3 1165.6 1232.0 1286.0	514.1 659.0 807.7 952.2 1085.9 1204.0 1306.7 1390.7	349.7 702.4 665.7 1028.6 1182.7 1322.3 144.6 1548.0 1633.6			
					Proo	Energy, Stu	16							
900 1000 1100 1200 1300 1400 1500 1630 1700	- 185.51 - 829.17 - 877.12 - 309.25 - 305.58 - 446.15 - 519.67 - 693.14 - 730.80		# 149.95 # 190.36 # 255.06 # 256.06 # 394.80 # 596.93 # 506.93 # 308.05 # 667.37	81.99 -e755-67 -275-25 -341-95 -442-70 -507-77 -577-19 -651.06	178.89 223,93 57.76 510,90 374.86 594.95 499.59 468.02 441.49	=177.90 =216.52 =216.15 =314.86 =370 16 =429.66 =499.61 =561.97 =654.80	- 176.39 - 216.09 - 262.18 - 511.96 - 560.73 - 423.75 - 409.23 - 557.19 - 629.64	=175.84 =215.94 =260.61 =309.75 =364.05 =465.75 =465.75 =467.65	- 174.22 - 214.32 - 256.91 - 507.89 - 561.03 - 400.05 - 400.79 - 550.06 - 621.90	-175.25 -213.19 -257.56 -306.27 -359.92 -117.63 -100.26 -347.24 -618.61	- 172.31 - 212.12 - 256.32 - 304.81 - 358.21 - 178.03 - 578.03 - 544.75 - 546.07			
					Ent.	py, Mtu/16/*	,							
900 1000 1100 1200 1300 1400 1500 1700 1800	,618 57 ,8709 ,90006 ,9410 ,58096 ,61968 ,69718 ,69758 ,72905 ,76348		37976 .42498 .44629 .51009 .55044 .56953 .66403 .6960 .79438	. \$1001 . \$5605 . \$9917 . \$403k . \$7998 . 61812 . 65508 . 69080	,30590 ,645,57 ,69068 ,5329 ,37329 ,61180 ,64911 ,68508 ,71993	. 57050 . 5379 . 64252 . 52652 . 56771 . 66008 . 68064 . 71366	.57544 .42706 .67589 .52103 .56298 .60964 .64051 .67695 .71214	. 37340 . 42591 . 47175 . 51677 . 53905 . 63717 . 67378 . 70912	.37189 .42178 .46897 .51363 .55589 .59601 .63429 .67104	. 37069 . 42019 . 46695 . 51128 . 55340 . 59352 . 65184 . 76867 . 70420	. 36970 . \$1,090 . \$6537 . 30944 . 55140 . 59145 . 66661 . 70218			

(Continued)

Table 104 (Contd-1). CAS FROPERTIES OF "SHELLDYNE"

Term.						Presnire, p	• (•				
Temp,	0	100	200	400	600	800	1900	1200	1Lug	1::00	1800
		, .		·	Viece	elty, lb/ft	/he				
-100 100 200 500 600 600 700 800 1100 1100 1100 1100 1100 1100	.0157 .011602160240024002500371 .0371 .0571	.0291 .0410 .0418 .0415 .0416 .0500 .0516 .0531 .0552 .0570	.042k .0440 .0457 .0472 .0490 .0507 .052k .0541 .0550 .0573		.0938 .0979 .097 1 .095 3 .095 9 .0578 .0579 .0592 .0603		.1474 .1092 .0943 .0725 .0474 .0447 .0447 .0453		.1786 .1427 .1147 .0968 .0781 .0781 .0775 .0710		.8011 .1651 1.990 .1178 .1096 .0965 .0061 .0796
] [,	,	Theres! Condu	etivity, Bu	1/05/55/55	,		,	, ———
-100 0 100 100 100 100 100 110	.9026 .C086 .0065 .0091 .0119 .0147 .020h .020h .0290 .0317 .0384 .0371 .0388 .0800 .0000	.02h5 .0252 .0346 .0346 .0379 .0422 .0453 .0466 .0467	.0995 .0322 .03-0 .0315 .0401 .0466 .0467 .0467		.0377 .0957 .0367 .0910 .0010 .0010 .0010 .0010 .0010		.04 50 .04 56 .04 50 .04 50 .04 50 .07 16 .07 16 .07 16 .07 51		.04% .0-\$2 .0+\$8 .0+71 .0-86 .0+22 .0-\$59 .0-\$59		.0470 .0469 .0473 .0497 .0497 .0398 .0349 .0349
1					Compress	ibility for	lor				
800 900 1000 1100 1200 1500 1400 1500 1600 1700 1800		.861 .908 .969 .945 .918 .918 .917 .961 .917 .990 .993	.800 .849 .869 .912 .934 .990 .962 .972 .980 .986		.516 .571 .799 .781 .861 .804 .917 .941		. 357 . 6 56 . 249 . 35 . 747 . 820 . 876 . 915 . 946		. hh 1 . hb 5 . j 5 h . j 5 h . 70 9 . b 5 h . 907 . 94 T		.991 .961 .665 .665 .730 .796 .857 .912
1					Heat Cope	10117, B:u/1	/*/*				
-100 0 100 900 500 500 600 700 600 1000 1100 1100 1100 1100 1100 1100 1100 1100 1100 1100 1100 1100 1100	.128 .128 .246 .503 .561 .412 .438 .500 .516 .510 .510 .625 .625 .636 .671 .691 .703 .714 .723	.584 .609 .654 .656 .676 .694 .705 .717 .725 .730	.626 .645 .662 .699 .709 .719 .727 .739	.702 .691 .699 .710 .717 .725 .731 .731	.949 .716 .729 .727 .728 .733 .731 .740	. 794 .054 .070 .746 .740 .741 .741	.765 .784 .781 .775 .724 .748 .748	•751 •765 •776 •776 •776 •775 •775 •775 •775	.744 .775 .765 .766 .762 .757 .757 .757	. 756 . 758 . 757 . 763 . 760 . 758 . 756	.734 .714 .773 .760 .750 .759 .758

(Continued)

Table 104 (Contd-2). GAS PROPERTIES OF "SHELLDYNE"

7000	Prosoure, pole													
1	0	100	P00	600	600	600	1000	1200	1400	1600	1800			
					Mont Cape	city Matio,	Cy/Cy			·····	<u> </u>			
900 1000 1100 1200 1300 1400 1500 1700 1700			1.039 1.032 1.007 1.004 1.009 1.001 1.019 1.019	1.055 1.052 1.059 1.052 1.028 1.026 1.024	1,505 1,129 1,066 1,046 1,039 1,035 1,029 1,027	1.138 1.173 1.109 1.069 1.051 1.051 1.053 1.051	1.100 1.121 1.114 1.094 1.063 1.061 1.041 1.036	1.009 1.099 1.099 1.099 1.069 1.055 1.040 1.040	1.075 1.081 1.005 1.076 1.068 1.058 1.049 1.053	1.068 1.072 1.073 1.071 1.065 1.058 1.051 1.044	1.069 1.065 1.065 1.069 1.074 1.076 1.076			
	Joule-Phomeon Coefficient, *7/pole													
900	<u> </u>		.1129	Γ	T	T	1	Υ	·					
1000 1100 1200 1300 7 20 1500 1600 1800			.0420 .0196 .0495 .0403 .0599 .0891 .0855	.1290 .0510 .0577 .0446 .0368 .0303 .0861 .0829	.1070 .1090 .4672 .0465 .0379 .3209 .0252	.0900 .067k .06k9 .0k8k .0976 .090k .0256	.0156 .0504 .0405 .0408 .0508 .0901 .0859	.0098 .0175 .0058 .0066 .0075 .0042 .0015	.006A .0110 .0160 .0196 .0196 .0197 .0181	.00h2 .007h .0108 .0137 .0155 .0155	.00% .0050 .0075 .0096 .0115 .0120			
7900			.0204	.0204	10807	.0194	.0183	.0166 7010.	.0165 .0149	.0140 .0130	.0117			
		Sound "elocity, ft/see												
900 1000 1100 1100 1100 1100 1600 1600 1			590. 597. 577. 610. 641. 668. 694. 718. 760.	996. 686. 940. 959. 664. 657. 607. 719.	309. 37h. 46p. 535. 50h. 60p. 66p. 65p.	946. 408. 436. 501. 537. 604. 640. 712.	676. 342. 686. 510. 333. 397. 636. 676.	777. 655. 577. 566. 571. 609. (AL. 678. 710.	Ma. 769. 664. 689. 681. 658. 664.	938. 825. 765. 695. 677. 690. 715. 739.	3006. 897. 818. 760. 731. 725. 729. 784.			

e) The reference state in the liquid at esturation pressure and O'F.

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